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JOURNAL OF

METALS

Vol. 13, No. 5
May, 1961

COVER: Argon teeming as an industrial technique for improving steel quality is the subject of two articles and David Cain's cover. Attention is also called to articles on beryllides and the role of bacteria in the leaching of copper ores.

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personnel

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Research Metallurgist, B.S. Met. Eng., age 42, married, 3 children. Fifteen years experience aluminum base alloys, free machining and high-strength alloy development, investigation of aluminum powder alloys and aluminum fibers, magnesium base alloys for forgings and elevated temperature use, process variables to improve magnesium base alloy; two years metallurgy of uranium and uranium base alloy; two years study oxidation of copper alloys, publications and patents available immediately. Prefer West but will relocate. M-250

Responsible Research Position desired. Qualified by eleven years fundamental and applied research, six years of university teaching and two years industrial experience at management level. Degrees in Met. and Physical Chem, Ph.D. '48. Thorough knowledge material science, flow and fracture of metals, phase transformations, physics of metals. Good knowledge solid state and electronics for possible integration into materials sciences in the semiconductor industry. M-251

Production Superintendent, BSEE, age 37. Ten years experience electric furnace operation producing alloys including six years as plant superintendent and higher. Duties included technical and operational direction and supervision, quality control and process evaluation. Location open. M-2130-Chicago

Physical Metallurgist, MS Met., age 30. Sound record of achievement in diversified fields of industrial research and development. Ability to organize and supervise programs and make clear written or oral reports. Any location. M-2131-Chicago

Metallurgical Engineer, graduate study, supervising laboratory, statistical quality control of raw materials, heat treatment, electroplating, mechanical properties, seeks permanent opportunity using more fully, judgement and 18 years experience in product development, troubleshooting, corrosion control, all metallurgical processes in avionics manufacturing and research. Prefer New York Metropolitan area. M-252

—POSITIONS OPEN—

Industrial Engineer, graduate industrial, chemical or metallurgical. Must have manufacturing know how, material movement, production scheduling, inventory and cost control for company manufacturing electrode rods, fluxes, etc. Future offers plant managership. $12,000-$15,000. New York, N. Y. W161

Spectrographer, B.S., preferably in metallurgy. Should have experience in emission spectrography. Will have to establish standard for routine analysis of copper, gold, similar and related by-products. Salary open. New Jersey. W151

Engineers, (a) Plant Superintendent, nonferrous metals and alloys, experienced in industrial relations, materials handling and familiar with furnace operation, gas and electric. Must be cost conscious. (b) Plant Metallurgist, non-ferrous metals and alloys, for a modern chemical and spectrographic laboratory; new product development; practical research work; to improve present processes. (c) Purchasing Assistant, nonferrous metals, secondary and fabricated. Technical and metallurgical background helpful. Salaries open. New York Metropolitan area. W126

Welding Engineers, degree in welding or metallurgical engineering, with 5 to 15 years experience, some of which should have been in the welding of stainless steel, (austenitic series), inconel, monel and carbon steel. Will be responsible for preparation of welding procedures and interpretation of specs, reports, etc. Salary to $12,000 year. W70(a)

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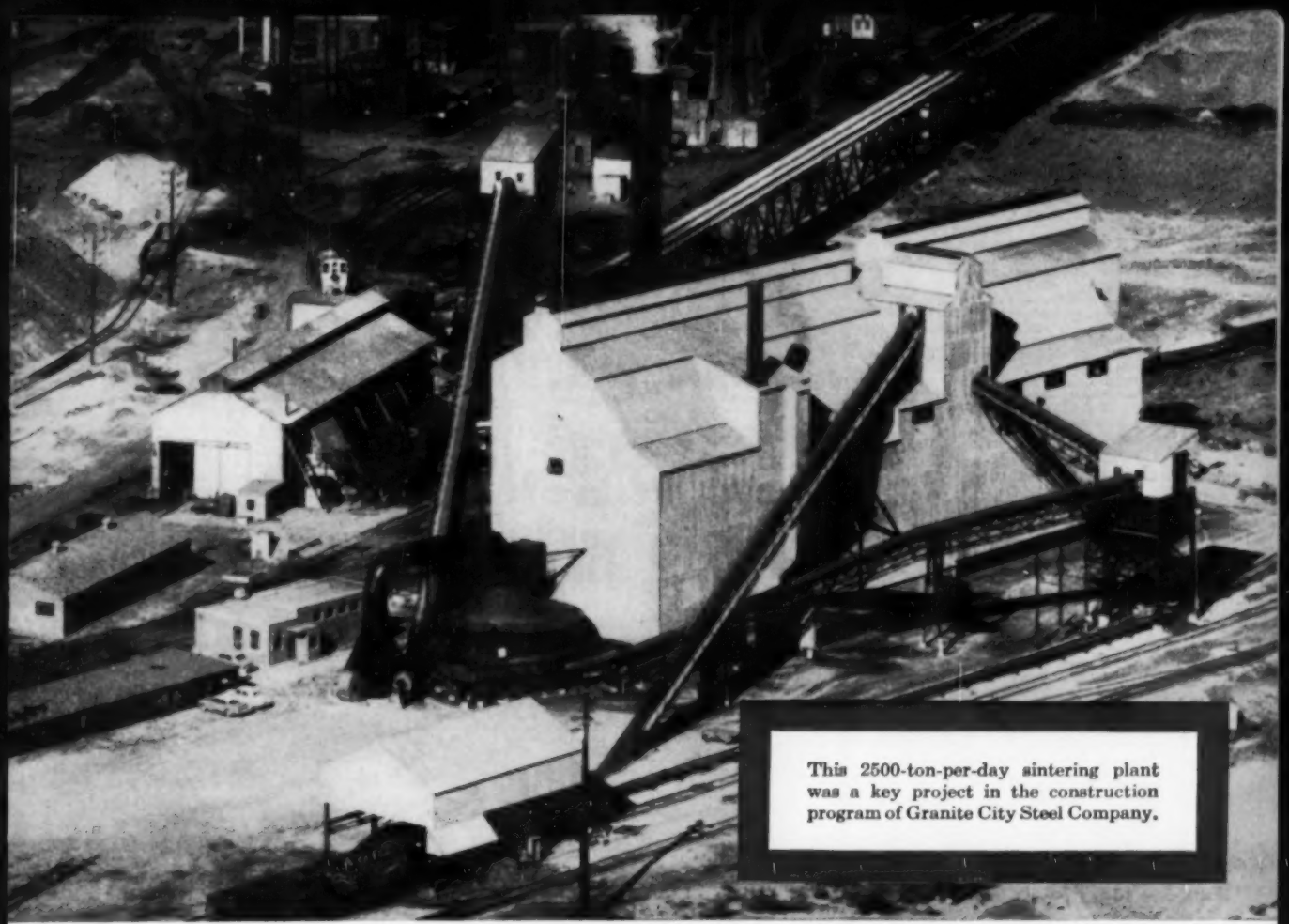

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personals

Anthony L. Ascik has been elected vice president of Tennessee Products and Chemical Corp. He joined the company in 1959 as director of metallurgical technical services.

Philip J. Berg has been appointed manager, sales and development, for the engineering and construction department of Dravo Corp.'s machinery div. **J. H. Porteus** has been appointed chief development engineer, ore processing.

William A. Bringman is now assistant superintendant of the masonry department of Kaiser Steel Corp's Fontana, Calif., plant. He was previously masonary engineer.

Ora E. Clark, general superintendent of Armco Steel Corp.'s Middletown works, will retire on June 1 after 34 years with the company.

Alvin G. Cook has been appointed coordinator, product specifications, of Allegheny Ludlum Steel Corp.'s Brackenridge, Pa., research center. He was previously assistant to the chief corporation metallurgist.

Theodore W. Cooper has joined Micro Systems, Inc., as manager of the operations div. He was formerly with Hughes Aircraft Co.

Emrys Davies, managing director of Brymbo Steel Works, has been awarded the Sir Robert Hadfield Medal for 1961 by the (British) Iron and Steel Institute.

Earl W. Donahue retired as works manager of the El Paso refinery of Phelps Dodge Refining Corp., effective March 31. **M. S. Bell,** assistant works manager has succeeded Mr. Donahue, and **H. A. Shaw** has become assistant works manager.

Herbert Fass is now general manager of Automation Alloys, Inc., Morocco, Ind. The company, which will produce and sell high-purity, precision semi-conductor alloys, was recently formed by Mr. Fass and several associates. He was formerly with Alloys Unlimited.

D. C. Hilty has been appointed assistant director of development in the technology department of Union Carbide Metals Co. He was formerly technical supervisor and manager, operations research.

Alan Lawley will be at the Franklin Institute in Philadelphia after May 22. He is presently at the University of Pennsylvania.

Roy O. McDuffie, of the University of Cincinnati's department of chemical and metallurgical engineering has been appointed the first Peter E. Rentschler professor of metallurgical engineering. The appointment is effective immediately.

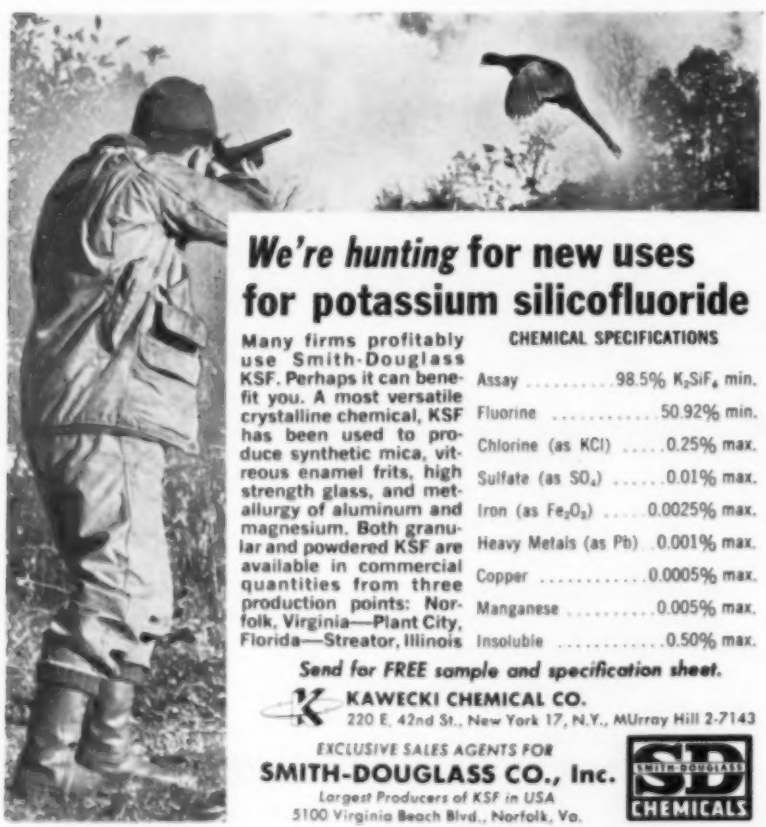
James D. Moore has been named manager of western operations for Vitro Chemical Co. He was formerly plant manager of the company's Salt Lake City plant.

W. S. Pellini, superintendent of the metallurgy div. at the US Naval Research Laboratory in Washington, has received the Navy Distinguished Civilian Service Award for work in the field of welding and brittle fracture. He has also been awarded the A.F.S. Penton Gold Medal Award for work in the field of casting and solidification of metals.

Walter C. Rueckel has been appointed vice president of Wilputte Coke Oven div. of Allied Chemical Corp. He was formerly with Koppers Co.

Walter M. Saunders, Jr. has been named metallurgical director of the Taft-Peirce Manufacturing Co., Woonsocket, R. I. Dr. Saunders is former owner and director of Walter M. Saunders, Inc., a commercial metallurgical laboratory in Providence.

Frank C. Senior is now vice president in charge of operations for Strategic Materials Corp. He has been general manager of the company's plant at Niagara Falls, Ont., since 1958.



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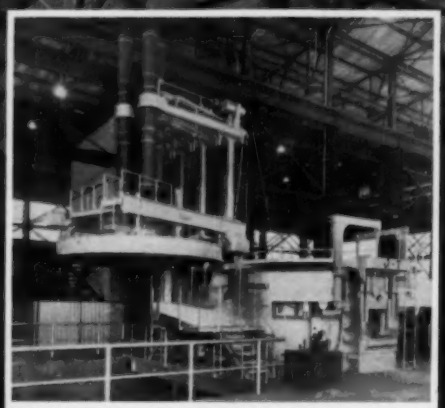
Any employer interested in receiving the bulletin should so advise E.S.P.S. at 8 W. 40th St., New York 18, N.Y., and your name will be placed on the mailing list. Any engineer who is registered with the Service or wishes to register is entitled to a 35-word notice in the bulletin. Write to the New York office for forms. Your qualifications will be brought to the attention of employers on our mailing list without revealing your identity.

It is planned to extend the publication to the midwestern and western offices in the near future.



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X-Ray Metallography

X-Ray Metallography, by A. Taylor, John Wiley & Sons, Inc., New York, 993 pp., $27.00, 1960. *Reviewed by A. S. Nowick, IBM Research Center.* •

This book apparently started out to be a new edition of the author's earlier *Introduction to X-Ray Metallography*, but in view of the advances of the last fifteen years, a completely new book was deemed necessary. Starting with the assumption that the reader has no prior knowledge of X-rays or crystallography and essentially no background in metal physics, the author attempts to cover the theory of X-ray diffraction and its application to almost every aspect of the study of metals and alloys. Included are the use of X-rays for the determination of crystal structures, of thermal equilibrium diagrams, of orientation textures, of grain size and internal stress, and for chemical analysis. Clearly, even in 993 pages, such a range of topics cannot be covered thoroughly. Nevertheless, almost every related topic is touched upon and extensive references are given. (In several of the larger chapters the number of references runs well over 100.)

As in every book, some readers will disagree with the relative amount of attention given to the various topics. In general, this reviewer found, with few exceptions, that the coverage was reasonably well balanced. The topic of the reciprocal lattice, however, which is confined to only one and a half pages is one such exception. In view of the assumed lack of background of the reader in metal physics, the author treats a number of maior subjects in extremely abbreviated form; among these are the thermodynamical theory of equilibrium diagrams, the theory of order-disorder, Brilloin zone theory, dislocation theory of plastic deformation, and the theory of the elasticity of crystals. Each of these topics is dealt with in only four to five pages, so that it is clear that the reader must actually acquire some further background to appreciate fully what may be learned about these subjects by means of X-ray techniques.

Some of the more unusual topics for a book of this kind are a full chapter devoted to radiography, microradiography and X-ray microscopy, and short treatments of the electron microprobe analyzer, microbeam methods, and the technique for direct observation of individual dislocations. The author occasionally steps out of the field of X-rays to touch briefly on electron and neutron diffraction as well as neutron radiography.

In spite of the diversity of topics the material is exceptionally well organized. (One exception is the topic of determination of the orientation of single crystals, which is found in the chapter under *Orientation Textures*. This may be convenient for presentation but it can easily be missed by a reader who is attempting to find material on the subject.) The book is rendered more useful by discussing commercial X-ray equipment throughout. Also, numerous tables of useful data pertinent to X-ray diffraction and crystallography are presented.

In summary, this book is not suitable as a textbook but can serve a useful function as a reference book. Unfortunately, the book is not priced to encourage beginners in the field of X-ray metallography, e.g., graduate students, to own it.

Introduction to Solids

Introduction to Solids by L. V. Azaroff, McGraw-Hill Book Co., Inc., 460 pp., $9.50, 1960. *Reviewed by J. B. Newkirk, Cornell University.* •

This book provides pleasant and instructive reading for the beginning student in solid state physics. It is logically organized and presented in a manner that marks its author as an experienced teacher. Also, mature metallurgists and engineers who wish to strengthen their knowledge in modern concepts of the solid state should find this volume well within their reach. Mathematical formulations are kept to a minimum and wherever possible new concepts and interrelations are pictorially or graphically illustrated. Stimulating problems follow each chapter and numerous basic references are given by which the reader can pursue his special interest.

Theories relating to the electronic structure of insulators, semiconductors and metals are carefully explained. However the structure and theory of the amorphous or glassy state are only superficially treated at the end of the book. Also, as if by contrast to the quality of the sections on crystallographic theory, crystallographic practice is seriously neglected. For example stereographic analysis is not mentioned, nor even the crystallographic reciprocal lattice.

Such vital metallographic items as Widmanstatten structure, precipitation phenomena, lineal analysis, sharp and diffuse diffraction effects are hardly mentioned. This situation is especially serious for the metallurgically oriented reader because several authoritative texts, where these matters are well treated, including the books of Cullity, A. Tayler, and Guinier, are nowhere given as references. Instruction in X-ray technology is not to be found.

In general this is a good text for introduction to modern solid state theory. It is not recommended as a practical aid in experimental metallography or crystal structure analysis.

Introduction to Ceramics

Introduction to Ceramics, by W. D. Kingery, John Wiley & Sons, Inc., New York, 781 pp., $15.00, 1960. *Reviewed by John D. Sullivan, Battelle Memorial Institute.* •

The ceramic schools and industry have long awaited a book such as Dr. Kingery prepared. Because this review is written primarily for metallurgists, stress is focused on facets that are useful to students, research and other workers in various fields of metallurgy, particularly physical metallurgy. Were the author teaching some phase of technology other than ceramics he probably would have written much the same book but would have changed its title and used different examples and illustrations. The book is on materials science with stress on ceramics.

It is based on a mimeographed form that has been used at Massachusetts Institute of Technology as the text for two subjects in ceramics. It is divided into five parts: I. Introduction, II. Ceramic Processes, III. Characteristics of Ceramic Solids, IV. Development of Microstructure in Ceramics and V. Properties of Ceramics. Parts III and IV form the basis of a one-term subject for undergraduate students who have had a previous introduction to physical metallurgy and thus are familiar with the first principles of crystal structure and phase diagrams. The material included in Part V forms the major part of a course on physical ceramics designed principally for graduate students. Part I (12 pages) and Part II (65 pages) are introduc-

(Continued on next page)

tory material, but are useful for an elementary course in ceramics. To metallurgists they are interesting reading as background material. Each chapter follows a pattern. It starts off with a brief introduction saying what the chapter covers, then gets into the presentation, followed by a list of suggested reading, and closes with problems.

The meat of the book is in Parts III, IV and V. The chapter title headings of Part III illustrates the scope covered: structures of crystals, noncrystalline solids, structural imperfections, surfaces and interfaces, and atomic mobility. A well-trained physical metallurgist is familiar with much of the subject matter of Part III but the author is owed a debt of gratitude for putting so much material together in a small space and for presenting it in a useful and readable manner. The book is profuse with illustrations and diagrams. In Chapter 4 on the structure of crystals this reviewer counted over 100 structure and similar models. This chapter is of as much interest to the metallurgist as to the ceramist. That on noncrystalline solids is of less use to the metallurgist, although some of it applies to slag systems. The chapter on structural imperfections applies equally well to metallurgy. In fact, much of the first work on dislocations was done in the field of physical metallurgy.

Part IV develops an understanding of the factors that determine the phase distribution and how they operate in ceramic systems. Chapter 9 has many phase diagrams. Although these are not new, some of them are of specific interest to metallurgists particularly those on the following systems: CaO-Al_2O_3, MgO-SiO_2, MgO-Al_2O_3, Ti-TiO_2, Fe-O, SiO_2-Al_2O_3, and MgO-Al_2O_3-SiO_2. The chapter on grain growth, sintering and vitrification, particularly that on the first two, is of interest to metallurgists.

Part V on properties of ceramics is broken down into seven chapters on thermal properties; optical properties; plastic deformation, viscous flow, and creep; elasticity and strength; thermal stresses; electrical conductivity; and dielectric and magnetic properties.

The author uses the mathematical approach in most chapters, and a mathematical background on the part of the reader is necessary to understand much of the book. Still there are many tables of data, both on ceramics and metals, that permit the book to serve as a handbook in some areas. Among the latter, the following are pointed out: In Part V among such data included in tabular or graphic form are mean thermal expansion coefficients, thermal resistivity, thermal conductivity of many ceramic materials, refractive indices of some glasses and crystals, glass-opacifier systems, creep, modulus of elasticity of some ceramics, electrical resistivity and dielectric constants. Another useful handbook-type table is given on page 344 on the composition of about 15 glazes and enamels. The triaxial diagram on the system silica-leucite-mullite on page 419 will interest many readers because it shows areas where many ceramic whitewares are formed. Likewise, the table on page 418 showing the composition of ten whiteware body compositions will be found useful. Many readers will find use of the table on pages 690 and 691 on conversion factors for electrical and magnetic units.

The quality of the book is such that minor errors and slips perhaps should be overlooked. It is only to be expected that there will be some in the first edition of a book nearly 800 pages long.

There is an excellent index, with considerable cross referencing, covering 21 pages. The printing of the book is good and the illustrations are clear. Photomicrographs are reproduced well on the quality of paper used. The book is a must for students of ceramics and it contains enough good information applicable to metallurgists that it is recommended for research and other workers in physical metallurgy. It is a useful book in any metallurgy library.

The Extrusion of Metals

The Extrusion of Metals by C. E. Pearson and R. N. Parkins, John Wiley & Sons, Inc., New York, 336 pp., $7.50, 1960, Second Edition. *Reviewed by Paul Lowenstein, Nuclear Metals, Inc.* •

It is a rather amazing fact that a field of such increasing importance as the extrusion of metals is covered only by a single book in the English language. This book, *The Extrusion of Metals* by C. E. Pearson, first appeared in 1944, was revised in 1953, and has now been revised a second time, in collaboration with R. N. Parkins. The lack of books published in this field is to a great extent offset by the very high quality of the Pearson book. In the latest edition the authors have changed relatively little compared to the first revision, but the extrusion technology itself has not changed drastically during these years. The authors have, in the meantime, gained a more thorough understanding of the difference between the two basic types of metal movement that occur in extrusion, streamlined flow and turbulent flow. They have also greatly strengthened the treatment of metallurgical aspects of the extrusion process. Many of the new materials and techniques developed in recent years are mentioned, and one new chapter dealing with the theoretical analysis of metal flow in extrusion has been added.

In the earlier edition, cable sheathing with lead was discussed in detail while sheathing with aluminum was only mentioned as a novelty. The latest edition gives considerable technical details about aluminum cable cladding.

Although the basic equipment for hot extrusion of metals has not changed, there is increasing emphasis today on equipment for higher speeds and high productivity. Such equipment is described, as well as methods of obtaining tapered solid sections and stepped extrusions. Modern heating facilities, especially for induction heating, are discussed.

The vital role of lubrication in determining the type of flow occurring in extrusion is recognized and the Ugine Sejournet glass lubrication process is discussed. Some very interesting recent work concerning the flow of metal in tube extrusions and in unsymmetrical sections is described.

The new chapter, devoted mainly to the theory of plasticity as applied to extrusion, is of great interest. The mathematical tools in support of plasticity theory have become more effective and solutions for many of the complex problems can be found. There may be some question concerning the immediate and practical gains in extrusion technology as the result of such work, but there is no question that it is through the route of plasticity that the extrusion process will eventually be transformed from an art to a more exact and controllable science.

The chapter on metals and alloys for hot extrusion has almost been doubled in size. The importance of speed in the extrusion of aluminum is recognized and discussed, and there is an expanded discussion of the extrusion of magnesium. Some mention is made of the extrusion of the newer metals, such as titanium, zirconium, molybdenum, and uranium. Special techniques such as fabrication of integrally clad fuel elements, extrusion of powders, extrusion of mixtures of metals and non-metals (such as the SAP process), and other special techniques are discussed.

In the chapter on treating of the properties of extruded metal, the point of view is metallurgical rather than mechanical; exaggerated grain growth, mechanical anisotropy, and preferred orientation are discussed in detail.

Some of the high velocity methods of metal extrusions and the effects of heat generation in high velocity extrusions are described. The chapter dealing with some special extrusion applications is greatly expanded and subjects such as the formation of intricate shapes by the filled billet method are discussed.

This new edition of *The Extrusion of Metals* can be considered a must for anyone in the extrusion industry.

(Continued on page 342)

education

Summer Program

Massachusetts Institute of Technology will present a special summer program on materials science from June 13-23 during the 1961 summer session.

The main theme of this special summer program is to present an over-all view of materials science in the light of recent teaching experience. It is intended as a course rather than a research conference. The lectures will be directed to men in industry, government, or universities who want to extend their knowledge of the fundamentals of materials to areas beyond their own specialties. Emphasis will be placed on the general interrelationships between the the fine structure of materials and their mechanical electrical magnetic and nuclear-radiation properties.

The program is under the direction of Professor Morris Cohen of the Department of Metallurgy and Prof. David J. Epstein of the Department of Electrical Engineering.

Tuition for the program is $325, payable upon notification of admission. No academic credit is offered.

Textbook Supplement

A new supplement for chemistry and physics textbooks is now available without cost, to high school science teachers. The 40-page manual, prepared by U. S. Steel Corp., and entitled Laboratory Experiments in the Chemistry and Physics of Steel, can also be used as review material for introductory chemistry and physics at first-year college level.

The main body of the spiral-bound, looseleaf booklet is divided into four parts: The Crystalline Nature of Metals; Physical Characteristics of Steel; Magnetic Characteristics of Steel; and Combatting Corrosion in Steel. Each part consists of experiments designed to acquaint the student with the physical and chemical laws that give steel its various characteristics.

Distribution will be limited to two copies for each instructor. Copies may be obtained by writing to the Public Relations Dept., U. S. Steel Corp., Room 1800, 71 Broadway, New York 6, N. Y.

Electron Microscopy

A two-week course in electron microscopy for research workers in the physical sciences will be given by the staff of the Electron Microscopy Laboratory of the College of Mineral Industries, Pennsylvania State University, University Park, Pa. The course will be given September 3-15.

The program will consist of lectures by the staff and personnel from industrial and government research laboratories. Topics covered will include basic theory of electron optics, instrumentation, electron diffraction, specimen preparation techniques, applications, and other subjects determined by the interests of the participants. Laboratory facilities are available for practical experience in techniques. Instruction will be given

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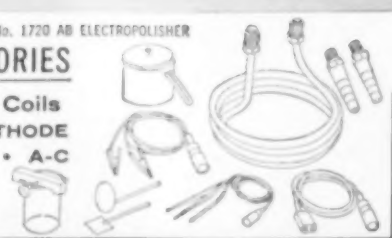

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AROUND THE SECTIONS

At a recent meeting of the Chicago section, AIME, the 1960 Robert Lansing Hardy Gold Medal was presented to Jerome B. Cohen, assistant professor at Northwestern University. Pictured, (l to r), are Mrs. Cohen, Dr. Cohen, and Carl H. Samans, who made the presentation.

Pittsburgh section's Institute of Metals Division met March 16 with Morris Cohen, professor of Metallurgy at Massachusetts Institute of Technology as the guest speaker. He discussed *Brittle Fractures in Mild Steel*. He said that the brittle fracture of mild steel at subzero temperatures remains one of the classical problems in physical metallurgy. The various theories of fracture were discussed in the light of recent experimental evidence. Systematic studies have been made on the role of metallurgical structure on brittle fracture, leading to further insight into micromechanisms of cleavage. The formation of microcracks before the propagation to complete rupture suggests a rational basis for distinguishing between crack initiation and crack growth.

Ohio Valley section's Nominating Committee has selected the following candidates to serve for 1960-61: H. B. Goodwin, chairman; V. D. Barth, vice-chairman; V. W. Ellzey, secretary-treasurer; R. L. Frantz, assistant secretary-treasurer; E. S. Bartlett, membership chairman; and J. E. Hanway, program chairman.

The new Nominating Committee is composed of J. W. Spretnak, chairman; G. H. Manning; D. E. Krause; H. R. Ogden; and J. R. Lucas.

Oregon section met February 22 with other engineering organizations in the Portland area to commemorate Engineer's Week. The meeting was held at the Benson Polytechnic School Auditorium. Glen DeKraker of Sangamo Electric Co., the speaker for the evening, discussed *Engineering—A Career of Opportunity*. He covered the various fields of engineering and tied the opportunities of each field to the space age, pointing out that today, in the field of engineering, the sky is the limit.

In conjunction with the meeting, a panel consisting of members from each engineering society was available for counseling service to the attending students. Two members of the Oregon section, Rudy Clark of Alcoa and Jens Thorbjornsen of Reynolds Metals Co., participated on the panel.

At a meeting on January 20, Hollis M. Dole, director of the Oregon State Department of Geology and Mineral Industries, reviewed the work of his Department during 1960 and discussed the projects covered by the various field parties. He stated that the extensive geological mapping done by the department over the past years has proved extremely valuable to companies and individuals seeking minerals for their work.

Chicago section, NOHC, held an All-Day Meeting March 8 at the Del Prado Hotel. Attendance was more than 300, a record for a Chicago section meeting. The morning session began with opening remarks by M. E. Nickel, chairman of the section.

There were technical sessions on *The Effect of Basic Furnace Operations on Furnace Refractories; Effect of Increased Open Hearth Furnace Productivity on Auxiliary Equipment;* and *Steelmaking Practices in the Far East and Western Europe.*

There were four technical sessions in the afternoon. The first was *Open Hearth and Blooming Mill Practices in the Manufacture of Seamless Tubing.* Other sessions were *Use of Exothermic Side Boards for Hot Topping Large Slab Molds; Open Hearth Quality Practices;* and *Study of Oxygen Roof Lance Practices in the Basic Open Hearth Processes.*

Following a cocktail hour, dinner was served in the Crystal Ballroom. Awards were given for the best papers presented. First place went to P. E. Dempsey for his paper, *Study of Oxygen Roof Lance Practices in the Basic Open Hearth Processes.* He received a $100 Savings Bond and a Certificate of Merit. Second place went to F. P. Johnson for his paper, *Effect of Increased Open Hearth Furnace Productivity on Auxiliary Equipment*, and third place went to O. J. Schiene and G. L. Michael for their paper on *The Effect of Basic Furnace Operations on Furnace Refractories.*

Chicago section held a split session meeting March 7 with approximately 80 members attending. Following a joint dinner hour, there were separate speakers for SME and The Metallurgical Society.

F. A. Dudderar of U. S. Steel Corp. was technical chairman of The Metallurgical Society session. A lecture entitled *Metal Failures Can be Explained* was delivered by Merrill A. Scheil of the A. O. Smith Corp. He discussed the various types of metal failure, such as fatigue, corrosion fatigue, stress corrosion, intergranular corrosion, and brittle fracture, as well as metal failure due to hydrogen.

NOTICE TO AUTHORS

Make sure that manuscripts are typed double-spaced before submitting them to the JOURNAL OF METALS. Failure to do so results in retyping, chance of error, and needless delay. To facilitate matters, submit papers in duplicate and enclose illustrative material such as drawings and glossy photos at the same time. Such illustrations should be suitable for reproduction.

Recently Accepted TRANSACTIONS Papers

The Gadolinium-Iron System, by V. F. Novy, R. C. Vickery, and E. V. Kleber (IMD)

The Gadolinium-Nickel System, by V. F. Novy, R. C. Vickery, and E. V. Kleber (IMD)

The Gadolinium-Cobalt System, by V. F. Novy, R. C. Vickery, and E. V. Kleber (IMD)

Dilatometric Investigation of Vacuum-Melted Zircaloy-2, by Josef Intrater (IMD)

Penetration of Liquid Bismuth into the Grain Boundaries of a Nickel Alloy, by R. F. Cheney, F. G. Hochgraf, and C. W. Spencer (IMD)

Notch Sensitivity of Refractory Metals, by A. G. Imgram, F. C. Holden, H. R. Ogden, and R. I. Jaffee (IMD)

The Solubility of Hydrogen in α-Iron, by M. L. Hill and E. W. Johnson (IMD)

The Melting of Malleable Nickel and Nickel Alloys, by C. G. Bieber and R. F. Decker (EMD)

The Thermal Decomposition of Cobalt Sulfate, by J. Stuart Warner (EMD)

Identification of Intermediate Phases in the Manganese-Titanium System, by R. M. Waterstrat (IMD)

Some Thermodynamic Properties of the Cadmium-Copper System, by Richard Borg (IMD)

The Solubility of Nitrogen in Solid Iron-Nickel Alloys Near 1000°C, by H. A. Wriedt and O. D. Gonzalez (ISD)

Vapor Pressure Studies on Iron and Chromium and Several Alloys of Iron, Chromium, and Aluminum, by E. A. Gulbransen and K. F. Andrew (IMD)

The Strain Rate and Temperature Dependence of the Yield Point in Mo in Torsion, by D. Weinstein, G. Sinclair, and C. Wert (IMD)

Creep of a Recrystallized Aluminum SAP-Type Alloy, by G. S. Ansell and F. V. Lenel (IMD)

A Study of the Growth of Voids in Copper during the Creep Process by Measurement of the Accompanying Change in Density, by Raymond C. Boettner and W. D. Robertson (IMD)

Orientation Relationships in the Heterogeneous Nucleation of Solid Lead from Liquid Lead, by B. E. Sundquist and L. F. Mondolfo (IMD-EMD)

Strengthening of Iron-Base Alloys Containing Columbium, by E. E. Underwood, E. M. Stein, and G. K. Manning (IMD)

The Heats of Solution in Liquid Tin of the Group III Elements Aluminum, Gallium, Indium, and Thallium, by J. B. Cohen, B. W. Howlett, and M. B. Bever (EMD)

Density-Pressure Relationships in Powder Compaction, by R. W. Heckel (IMD)

The Effect of Hydrostatic Pressure on Self-Diffusion in Lead, by John B. Hudson and Robert E. Hoffman (IMD)

Determination of Number of Particles Per Unit Volume from Measurements Made on Random Plane Section: The General Cylinder and the Ellipsoid, by R. T. DeHoff and F. N. Rhines (IMD)

On the Intersection Mechanism of Plastic Deformation in Aluminum Single Crystals, by S. K. Mitra, P. W. Osborne, and J. E. Dorn (IMD)

Dynamic Effects During Twinning in Alpha Iron, by Erhard Hornbogen (IMD)

The Effect of Striation Substructure on the Critical Resolved Shear Stress of Zinc Single Crystals, by J. Rezek and G. B. Craig (IMD)

Recrystallization of Iron and Iron-Manganese Alloys, by W. C. Leslie, F. Plecity, and J. T. Michalak (IMD)

Thermodynamics and Kinetics of the Deoxidation of Thorium by Calcium, by David T. Peterson (EMD)

The Evolution of Textures in FCC Metals, Part II: Alloys of Copper with Phosphorus, Arsenic, and Antimony, by R. H. Richman and Y. C. Liu (IMD)

Metal Deposition Coefficients in Filament Bundles, by J. H. Oxley, J. E. Oberle, C. E. Dryden, and G. H. Kesler (IMD)

The Growth of Large Single-Crystals of 99.9% Iron of Controlled Orientation, by D. F. Stein and J. R. Low, Jr. (IMD)

Embrittlement of NaCl by Surface Compound Formation, by W. H. Class, E. S. Machlin, and G. T. Murray (IMD)

Annealing Twins and Coincidence Site Boundaries in Zone-Refined Aluminum, K. T. Aust (IMD)

Density of Liquid Iron Silicates, by John Henderson, R. G. Hudson, R. G. Ward, and G. Derge (ISD)

A Reinvestigation of the System Ti_2Cr and Ti-V, by Felix Ermanis, Paul A. Farrar, and Harold Margolin (IMD)

Technique for Determining Orientation Relationships and Interfacial Planes in Polyphase Alloys-Application to Controlled Eutectic Specimen, by R. W. Kraft (IMD)

The Effects of Interstitial Solute Atoms on the Fatigue Limit Behavior of Titanium, by Harry A. Lipsitt and Douglas Y. Wang (IMD)

Quantity and Form of Carbides in Austenitic and Precipitation Hardening Stainless Steels, by R. J. Bendure, L. C. Ikenberry, and J. H. Waxweiler (IMD)

The Control of Annealing Texture by Precipitation in Cold-Rolled Iron, by W. C. Leslie (IMD)

Recrystallization of a Silicon-Iron Crystal as Observed by Transmission Electron Microscopy, by Hsun Hu and A. Szirmae (IMD)

The Thermal Diffusion of Hydrogen in Alpha-Delta Zircaloy-2, by J. M. Markowitz (IMD)

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First Kaldo Plant in USA!

Sharon Steel Corp., will become the first company in the U.S. to install Stora Kaldo furnaces. The company expects to tap the first heat from the new facility, which will be built at the Roemer works at Farrell, Pa., in the fall of 1962.

Dravo Corp. will design and build the $17.5 million plant, and the National Cylinder Gas div. of Chemetron Corp. has been awarded a contract by Sharon to build and operate an on-site air separation plant to supply 370 tpd of high-purity oxygen for the process.

The two-furnace Stora Kaldo installation, tapping 150-ton heats, will have an initial capacity of approx 1 million ingot tons annually. Provisions are made in the design of the plant for an additional furnace that could double this output.

Prof. Bo Kalling, inventor of the process, will serve as consultant to Sharon and Dravo during the construction and early operation of the new furnaces.

The new melt shop, complete with charging, furnace and teeming aisles, along with scrap and slag handling bay, will be housed in a building 500 x 244 ft. The plant has been designed to expedite the movement of raw materials and finished steel ingots. The furnaces, 25 ft long and 18 ft in diam at the center, will rotate in a cradle at speeds varying up to 27 rpm, and an angle of 17°. High-purity oxygen is to be injected into the vessel at 20 to 25 psig through a water-cooled oscillating lance mounted in the movable exhaust hood which fits over the mouth of the furnace. By using wet scrubbers in the dust collection system, air pollution will be closely controlled.

According to James A. Roemer, Sharon's board chairman, "As a result of carefully controlled quality tests over the full range of carbon steel analyses produced in our open hearth furnaces and in electric, LD, and Stora Kaldo furnaces, we concluded that steel made by the Stora Kaldo method would best supplement our present range of specialty steels."

Li-Mg alloys

Li-Mg alloys, 20 pct lighter than conventional magnesium alloys, are potentially useful in missile and space structures, according to a report prepared by Battelle Memorial Institute for the Army Ballistic Missile Agency.

Reducing the weight of space vehicle structures is critically important because of the high ratio of booster weight to payload—up to 1000 lb of rocket and fuel for every pound of payload put into space. Light structural components in the satellite and upper stages of the rocket greatly increase the efficiency of rocket system, permitting larger, more fully instrumented satellites to be orbited.

Magnesium-lithium alloys appear to be the lightest metals suitable for structural use; this means that they can provide stiffer structures for less weight. Although not having especially high strength—about 25,000 psi—the alloys should be valuable in space structures which must be light but need not carry heavy loads.

USSR converter steel

An extensive program of construction of powerful converters has been adopted in the USSR. When completed, the share of converter steel in total Soviet production will be tripled within the next five years to reach almost 20 pct by 1965.

A standard design for a 2 to 4 million ton converter shop has been worked out at the Moscow Institute for Designing Steel Works. Three [probably oxygen,—Ed.] converters of 100 to 130 tons' capacity will each produce some 2½ times as much steel a year as the biggest open hearth furnaces at a much lower capital cost. Converters with a capacity of 200 to 250 tons are now in the design stage. These shops will be constructed at Nizhniy Tagil, Lipetsk, Chelyabinsk, and other sites.

Integrated Ni production

International Nickel Co. of Canada, Ltd. has begun operation of the world's first fully integrated nickel-producing facility at Thompson, Manitoba, Canada. Capacity is estimated at 75 million lb of electrolytically refined nickel per year. The $185 million project is expected to raise Inco's annual capacity for producing nickel metal to over 385-million lb.

In addition to the mine itself, facilities include a concentrator; a smelter consisting of fluid-bed roasters, electric matte furnaces, and converters; and a refinery where converter metal cast into anodes is refined electrolytically. Power is supplied by the Manitoba Hydro-Electric Board plant which is located some 50 mi from the Inco site.

New Ti casting method

A new method caller *Impel Casting*, developed by Titanium Metals Corp. of America, has introduced titanium to the permanent mold, resulting in a new, low-cost method for casting small parts in production quantities. With graphite molds, the most popular method employed for titanium casting up to the present, production was limited to 10 to 12 castings per die, whereas the new method is said to be able to produce up to 100 castings per mold. TMCA has already produced 40 castings per mold.

In the new process, molten titanium is obtained from a double-melted titanium electrode, and then impelled into mild steel molds. Because of the shrinkage rate of titanium, feeding times are critical and must be controlled carefully. In general, properties to those of bar products have been obtained with the new process. Commercially-pure grades of titanium are being cast where corrosion resistance is more important than extremely high strengths.

REPORTER

Olin alumina process

Olin Mathieson Chemical Corp. recently announced the development of a process for the extraction of metal-grade alumina from domestic aluminum silicate ores which are abundant throughout the US. The announcement was made during the dedication of Olin's $7.5-million research center at New Haven, Conn. The process —still in the pilot-plant stage—consists of sulfuric-acid leaching the ores containing as little as 20 pct alumina, separating the aluminum sulfate from the liquor, and processing the resulting crystals into alumina.

The first step in the process is a normal leaching operation, but the crystallization step is significantly different from similar processes. In the other processes, aluminum sulfate forms tiny, soft crystals which are difficult to separate from the liquor, whereas the Olin process produces large, easily-separated crystals. The third step in the process is the thermal decomposition of the aluminum sulfate crystals into alumina and the recovery of sulfuric acid for re-use in the leaching operation. Several methods have been evaluated in the laboratory and will be tested with the pilot-plant equipment. This alumina-from-clay process should be particularly attractive to aluminum producers in areas where coal is available for cheap electric power and shales are abundant as coal mine waste.

Canadian iron plant

The first iron to be made in western Canada from local ores will be produced at the new pig iron plant of Consolidated Mining & Smelting Co., at Kimberley, B. C. The $7.5-million installation, consisting of a sintering plant and an electric furnace plant, has a capacity of 100 tpd. The plant is the initial stage of a major iron and steel production project at Kimberley which is expected to produce 300 tpd of pig iron, steel ingots, and basic steel products. Estimated cost of the entire project is more than $20 million.

Iron is produced from the western ore reserves of Cominco's Sullivan mine at Kimberley where lead and zinc content of the ore has been recovered for years, resulting in a large stockpile of iron-containing ore. There is a current stockpile of 15 million tons of iron concentrate and present lead and zinc refining operations will produce more than 350,000 additional tons per year.

Refractory micropowders

Uniform micropowders of refractory compounds are being produced at Linde Co.'s research laboratories. Columbium carbide and titanium carbide powders, less than 44 microns in size and of homogeneous composition, have been made and are available as 1 oz research samples. Linde began producing spherical powders in 1960.

The micropowders do not have the characteristic sphericity of the Linde refractory metal microspheres, but they are solid, uniform in particle size and composition, and do not agglomerate. It is expected that this proprietary process may be extended to include particles up to 1 mm in size.

Beryllium ore processes

Two companies—United Technical Industries, Inc., and Mineral Concentration Chemical Co.—have recently announced processes for the concentration of domestic low-grade beryllium ores. The UTI process will be performed at Delta and Salt Lake City, Utah and will utilize ion exchange and chemical processing to convert the ores to high-purity beryllium oxide. The operation's capacity is expected to be some 250 lb per day.

Mineral Concentration & Chemical Co.'s process—known as the *Mincon* process—is a heat flotation method of extraction. The Loveland and Berthoud plants of the company are now producing beryllium oxide with over 90 pct beryllium recovery. The beryllium oxide product ranges in purity from 96+ to 99+ pct BeO, according to company sources.

In addition to these companies, Beryllium Resources, Inc. (see the February 1961 issue of JOURNAL OF METALS, p. 139), Brush Beryllium Corp., and Vitro Minerals Co. are other American companies interested in concentrating low-grade US ores.

High-Temp X-ray spectrograph

The US Bureau of Mines has developed a fluorescent X-ray spectrograph which can study surface chemistry of molten metal while it is actually oxidizing at temperatures of 1650°F.

Using a resistance-wound electric furnace as a sample holder, the instrument brings the molten surface being studied within ½ in. of the X-ray tube window. Scientists at the Bureau of Mines' Metallurgy Research Center at College Park, Md., who developed the spectrograph, will use it as the basic tool in dynamic oxidation rate studies of base metals.

The spectrograph is said to be a progressive step toward equipment capable of close-up studies of materials at temperatures up to 2500°F. Such equipment would make possible new areas of research in fused salts and molten metals for space age technology.

Thin tinplate

At least three other companies will enter the thin tinplate field during 1961. Joining U.S. Steel Corp. as producers will be Jones & Laughlin Steel Co., Bethlehem Steel Co., and Wheeling Steel Corp. Others known to be working along these lines include Weirton Steel div. of National Steel Corp., Granite City Steel Co., and Youngstown Sheet & Tube Co.

Extra light, thin tinplate is produced by putting standard gage tinplate through a second rolling operation after the tin coating has been applied. This second rolling reduces the thickness of both the steel and the coating approx 50 pct. Weight reduction and consequent cost reduction are important factors in the unquestionably growing demands for the product. Thin tinplate is expected to compete actively with aluminum in the container market.

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Books

(Continued from page 333)

X-Ray Microscopy

X-Ray Microscopy by V. E. Cosslett and W. C. Nixon, New York: Cambridge University Press, $15.00, 1961. *Reviewed by Sigmund Weissmann, Rutgers University.* •

X-ray microscopy and microanalysis by X-rays have only recently become practical and important research tools. The excellent book by Cosslett and Nixon gives a clear account of the principles, applications and limitations of the various techniques and should be welcomed by students and researchers alike who are interested in this field.

The first chapter introduces the reader briefly to the various methods of X-rays microscopy and microanalysis and to the application of the methods in biology and inorganic specimens. This introductory chapter forms the nucleus for almost all subsequent chapters in which the various enunciated topics are developed in greater detail.

Chapter two covers the method of contact microradiography which has been developed by Engström and his school as a powerful method of microanalysis in biology and particularly cytology.

The third chapter deals with microscopy by point projection, and the effective development of this method is principally associated with the authors themselves. Chapters 4 and 5 deal with reflection X-ray microscopy and Chapter 6 with X-ray absorption microanalysis. Chapter 7 gives a comprehensive account of the recent developments in X-ray emission microanalysis and particularly the latest developments of the static and scanning microprobe analyzer are well presented.

The subsequent chapters are devoted to practical details and application in biology, medicine, metallurgy and technology.

Unfortunately, various diffraction methods which have recently become important in the study of lattice defects such as the Lang X-ray topography, the Borrmann method for studying dislocations and recent developments of the divergent beam method found only very brief mention. This neglect, however, is a measure of the rapidity with which the subject continues to develop rather than a defect in the work.

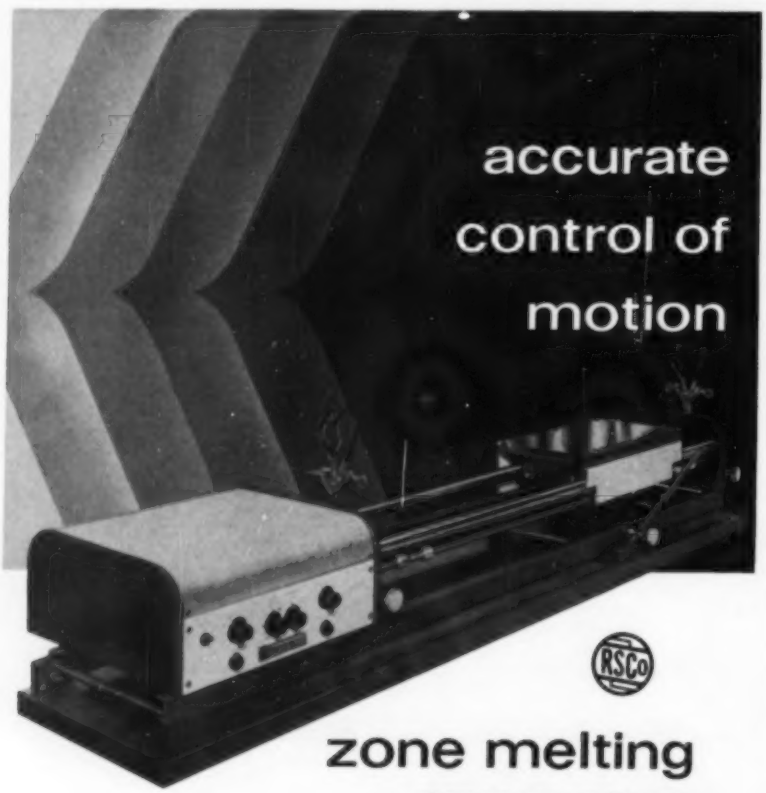
The book is adequately illustrated with line diagrams and well-reproduced micrographs. The appendix contains absorption and emission data.

• • •

Long Term Trends and Problems of the European Steel Industry, published by the United Nations' Economic Commission for Europe (ECE), Geneva, Switzerland. Distributed by Columbia University Press, 76 pp., $2.00, 1960—Based on an extensive study begun in 1956, this publication examines world consumption and production of steel and steelmaking raw materials and the economic consequences of technical trends, past and future. The final three chapters assess the future of steel in relation to European requirements, discussing possible solutions to some of the problems raised by the study, and indicating the nature of others. ECE cautions that all long-range forecasts need to be examined periodically, but adds that the comprehensive statistics here assembled should be universally useful.

Symposium on Newer Metals, ASTM Special Technical Publication No. 272, 218 pp., $7.25, 1960—The three sections of this text correspond to the three sessions of the Symposium. Section 1, Properties of Refractory Metals, includes papers on high-temperature testing methods and properties of molybdenum, tantalum, columbium, and the platinum-group metals. Section 2, Nuclear and Light Metals, includes papers on beryllium, zirconium, and yttrium. Section 3, Processing of Newer Metals, is concerned with purification and fabrication of columbium, tantalum, molybdenum, chromium, and lithium.

(Continued on page 386)



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editorial

Extractive Metallurgy — 1970

[In the following condensation of his Foreword to the recently published conference proceedings—Extractive Metallurgy of Copper, Nickel and Cobalt—the editor, Paul Queneau, provides us with an interesting insight into the state of Extractive Metallurgy in 1970.]

The vast, low-cost hydro-energy potential present in many remote regions of the world, and the impact of nuclear-power evolution will lead to increasing utilization of electric furnaces in the coming decade. Direct use of nuclear heat to raise the temperature of process reactants to above 3000°F will loom large on the horizon with inadequate materials of construction athwart its path. Improved furnace designs will be employed for utilization of oxygen in continuous autogenous smelting and converting. The tuyereless, top-blown oxygen steel converter will invade and conquer the smelters and refineries of the nonferrous industry. Fluid bed systems compound separation by controlled cooling, and purification by zone refining will play an expanding role.

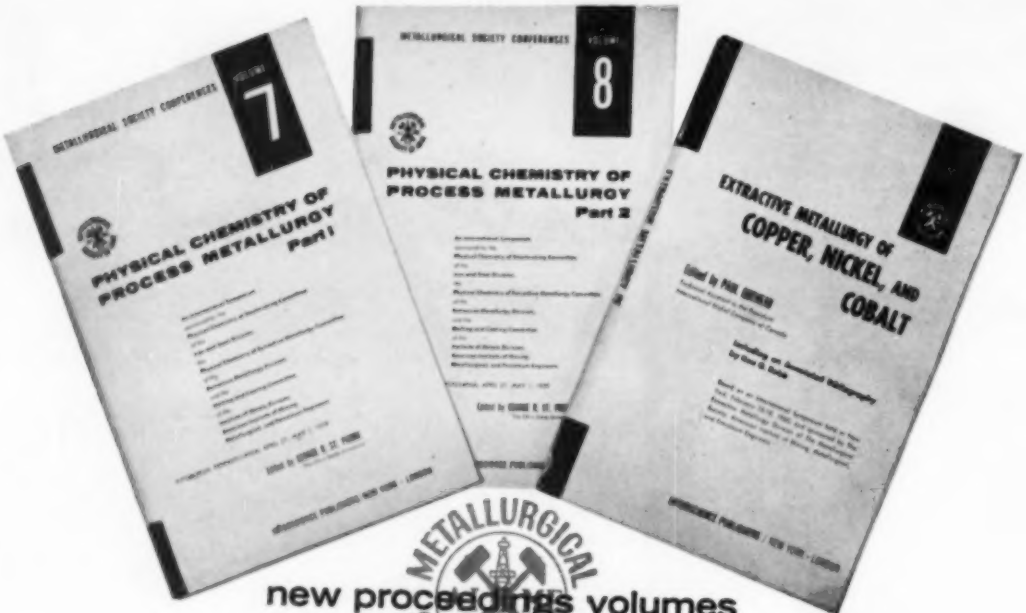
The marked trend toward hydrometallurgical processes in recent years will continue. Analytical techniques based on solvent extraction, ion exchange, and chromatography have commercial capabilities for separations on a large scale which will become increasingly apparent. Higher ground will be attained in fused salt electrolysis, in the separation of elements in complex molten solutions by continuous liquid-liquid extraction, and in the application of fractional distillation and selective vacuum volatilization.

An arena of metallurgical endeavor which will assume greater prominence is that of vapor phase reactions, for which I have coined the term *vapometallurgy*. When the extractant is a gas, such as a halogen or carbon monoxide, panoramic thermodynamic and kinetic vistas are opened up. Sophisticated processes will be designed in which amenable desired elements in the solids being treated will be converted into new compounds as solids, liquids, or gases and then in turn isolated as liquids or gases from which pure metals and the original gaseous reactant will be recovered.

There will be increasing use of pressures well above and below those now generally considered practical. Cryogenics will become a familiar term, and the alert engineer will detect the warming glow of the high temperatures of plasma vapometallurgy. The forward movement of extractive metallurgy in the direction of programmed and monitored continuous procedures will be hastened by continued rapid advances in laboratory and plant instrumentation, including the computer and radiation counter.

But a plea must be made for much greater research effort by the nonferrous extractive industry, which has unduly low research and development expenditures devoted to the recovery of metal from rock and from an important metal source of the future—scrap. This situation cannot long endure in the face of encroachments on metal territory by non-metals, of decreasing quantity and quality of ore reserves on this continent, and of increasing activity by totalitarian state monopolies. Ores are considered to be non-renewable resources, but the definition of the word ore is flexible and depends on many factors, not the least being the degree of seed investment in research and development today so that there will be a harvest to reap tomorrow. Essential to the future health of any major metal producing organization will be appropriately staffed and instrumented central and plant laboratories and pilot-plant facilities, adequate professional cadres at the operating level, and understanding policies in respect to technical personnel. The institutions of higher education, founts of trained manpower and virgin scientific knowledge, need and deserve generous support from their mining industry beneficiaries. These several requirements can and must be boldly met.

Paul Queneau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ARGON CASTING FOR IMPROVING STEEL QUALITY

Tests have shown that complete argon atmosphere protection of molten steel during ingot casting results in a 30 to 65 pct reduction in non-metallic inclusions, a decrease of 3 to 24 pct in conditioning losses, and a preservation of the effects of vacuum treatment.

by M. F. Hoffman, P. G. Bailey, and R. L. W. Holmes

Cleanliness of metals and their dissolved gas contents have become increasingly important to metal producers and users. As the physical property requirements of high-quality metals and alloys become more and more stringent, increased attention must be devoted to non-metallic inclusions and dissolved gases which adversely affect these physical properties. Measurable progress has been made in attaining higher quality product by close control of melting practice and by special treatments. However, in most processing practices the molten metal is exposed to air in the final step of casting ingots. This exposure to air can be eliminated by argon casting, now developed as an economical and practical technique for providing inert gas protection to molten metal during casting. This process has proven beneficial in protecting aircraft quality, forging grade, stainless, and specialty steels and nickel-base alloys. With ladle vacuum-treated steels the effcts of the degassing treatment are preserved.

Recent literature[1-5] points out that a large portion of the non-metallic inclusions existing in the ingot have their origin in the teeming operation. This is shown by radioactive tracer studies which conclude that only 4 to 18 pct of ingot inclusions come from ladle and pouring refractory erosion. It is believed that the teem stream contributes a much greater portion of non-metallic inclusions because of the large surface area exposed to oxidation. A ton of steel passing through a 1½ in. diam nozzle as a liquid cylinder has 144 sq ft of surface area exposed in the ladle to mold stream. Actually the falling stream does not remain a cylinder but tends to break up thereby exposing an even greater surface area. As the metal fills the mold, the oxides formed by exposure to air are dispersed by the metal agitation. Metal temperature in the mold decreases rapidly thereby increasing the viscosity of the metal. Because of this increased viscosity and because of the short time between teem and actual solidification, the inclusions are unable to float to the surface and are trapped in the body of the ingot. Argon protection of the teem stream prevents these oxides from forming.

In addition, the argon atmosphere in the mold protects molten metal splash from oxidation thus allowing the metal droplets adhering to the mold wall to remelt into the rising metal. The occurrence of scabs and laps is thus reduced. The oxide surface formed on droplets during air casting slows heat transfer across the droplet surface and impedes remelting of the droplet. Reduction in droplet oxidation also reduces the occurrence of hot working cracks at non-metallic discontinuities in the ingot surface. Increased metal yield and reduced conditioning costs can thus be realized.

Vacuum treatment is used to reduce dissolved hydrogen, oxygen, and nitrogen in steels. When vacuum-treated metal is teemed in air some of the benefits of the treatment are lost by exposure to the atmospheric gases. Unless adequate protection is provided, the high metal temperature, large exposed molten-metal surfaces, and the low gas content of the treated metal provide a strong driving force towards resolution of atmospheric gases. The protection provided by argon casting thus becomes desirable in the handling of vacuum-treated metal.

Process

Argon casting provides complete inert-atmosphere protection of the metal throughout the casting operation. Two distinct steps—mold purging and teem stream shrouding—are involved. Purging the mold insures inert or protected starting conditions. Shrouding the teem stream with argon maintains an inert atmosphere during the filling of the mold and protects the high surface area teem stream from ladle to mold. Previous attempts at argon-protected casting usually used either mold purging or stream shrouding but not both. A sacrifice in atmosphere quality or an excessive consumption of argon re-

M. F. HOFFMAN, P. G. BAILEY, and R. L. W. HOLMES are with the Newark Development Laboratory of Linde Co., a div. of Union Carbide Corp. This paper was presented at the Eighteenth Annual Electric Furnace Conference.

sulted. Included among these early procedures were lance pipe purging of uncovered and unsealed molds and directing jets of argon through air toward the metal stream. Development of the process described in this paper started out in much the same manner, but the use of an oxygen analyzer for mold atmosphere monitoring indicated the inadequacy of these approaches.

Two requirements must be fulfilled in establishing a truly inert atmosphere effectively and efficiently: 1) special attention must be directed toward mold preparation so that the mold cavity is made semi-gas-tight to minimize the possibility of argon leakage and dilution of the inert atmosphere by air currents passing over the mouth of the mold and 2) introduction of the purge argon should be in such a manner that a displacement rather than a dilution purge is achieved. The first requirement is satisfied by covering the mold and sealing the hot-top joint. The second requirement is met by using an appropriately designed purge gas distributor.

Argon is particularly effective as a purge gas because it is 38 pct heavier than air. Displacement purging, to be described later, cannot be practiced effectively with helium or nitrogen. Helium is extremely difficult to contain in the mold cavity because of its extreme lightness and because of its high diffusivity. Nitrogen with a density very nearly that of air will not displace air and can only be used to purge by diluting the air in the mold.

Mold preparation

Because of argon's density some attention is needed to insure a seal at the mold bottom. With big-end-up molds it has been found that the plug in the bottom usually fits tight enough to eliminate the necessity for added sealing. With big-end-down molds, some auxiliary sealing in the form of refractory mud or asbestos rope is usually required at the mold-stool joint.

Mold-hot top joints must be sealed to prevent air currents flowing through these joints and contaminating the inert gas atmosphere. Tiger hot tops and cast hot tops resting on the top of the mold, where the joint is sealed with refractory mud, require no special treatment. With C&D hot tops, asbestos rope is wrapped around the hot top below the wiper. As the hot top is set in place, the rope forces the wiper tightly into the gap thereby forming a satisfactory seal. It has been found with asbestos rope sealing of C&D hot tops that molds may be teemed all the way without a pause to allow the metal to solidify at the mold-hot top joint.

Table I. Nominal Composition of Argon Cast Steels

Steel	Alloy Elements, Pct								
	C	Cr	Ni	Mn	Si	Mo	P	S	Others
Suprimpacto	0.12	1.50	3.75	0.50	0.20	—	0.030 Max	0.030 Max	—
SAE-8745	0.45	0.50	0.55	0.80	0.30	0.25	0.040 Max	0.040 Max	—
SAE-4130	0.30	0.95	—	0.50	0.30	0.20	0.040 Max	0.040 Max	—
AHT 28	0.30	1.30	4.00	0.50	0.25	0.20	0.030	0.030	Al 0.02
Modified 4340	0.40	0.75	1.75	0.75	0.30	0.40	0.040 Max	0.040 Max	V 0.20
Nitralloy N	0.25	1.20	3.50	0.55	0.30	0.25	0.040 Max	0.040 Max	Al 1.25
SAD 155	0.20	0.60	2.50	0.60	0.20	0.60	—	—	V 0.20

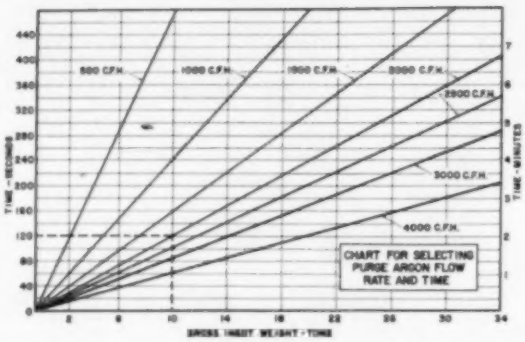


Fig. 1—Purging times at various flow rates as a function of ingot weight.

Brick hot tops must be sealed after they have been set on the mold because they are much closer fitting and more fragile than other types of hot tops. In this case, the asbestos rope is tamped into the space between the mold and the hot top. Experience with brick hot tops has not been extensive enough to determine if the ingot can be poured all the way without a pause.

To complete sealing of the mold chamber, the hot top opening must be covered. The most satisfactory sealing has been obtained with 0.003 in. thick aluminum foil. Domestic or kitchen foil, which is only 0.0015 in. thick, is too thin and delicate. A piece of foil 3 to 6 in. larger than the outer dimensions of the hot top should be used as a cover for the opening. The foil can be held in place by a split ring or by folding down and wiring to the hot top. The split ring allows lifting the foil cover to admit the purge gas distributor before purging. When the foil cover is wired, access for the gas distributor is provided by piercing a hole in the cover. This hole is taped shut again after purging is completed. Previously prepared patches, taped or glued to the covering, facilitate the resealing.

Purging

A completely inert atmosphere surrounding the teeming operation cannot be attained without first establishing an inert atmosphere in the mold cavity. Experience has shown that reasonable teem protection without prior mold purging only partially eliminates air from the mold.

Purging in the argon casting process takes advantage of argon's greater-than-air density to establish an inert atmosphere most economically in the mold cavity. Purging can be accomplished by dilution or displacement. Dilution purging is the most wasteful and uneconomical way of eliminating air from the mold. The purge gas, introduced through a straight pipe at high velocity, thoroughly mixes with the chamber atmosphere, and a portion of the purge gas is exhausted along with the ambient chamber atmosphere. With a dilution purge, 7 to 10 mold cavity volumes of purge gas are required to reduce the oxygen content in the chamber to less than 0.25 pct.

Displacement purging was developed specifically for the argon casting process described in this paper. Displacement purging with heavier-than-air argon, introduced at the bottom of the mold, forms an upward moving front much like a piston, pushing the mold atmosphere out of the top of the mold. The ideal displacement purge, therefore,

would require only 1 mold volume to establish a completely inert atmosphere. However, due to gaseous diffusion at the mold atmosphere interface, slightly more than 1 chamber volume of the purge gas is required to establish an inert atmosphere. In the recommended argon casting process, 1.5 to 1.75 mold volumes of argon are used to obtain less than 0.25 pct O_2 in the atmosphere at the mold-hot top joint. This is the most economical method for obtaining an inert atmosphere in the mold.

Experience in laboratory tests and in mold purging has shown that purging efficiency is practically independent of gas flow rate with a properly-designed purge gas distributor. Therefore, to minimize the holding time between purging and teeming, the purging operation can be synchronized with teeming so that one mold can be purged while the previous mold is being teemed. Fig. 1 shows purging times at various flow rates as a function of ingot weight. This diagram has been prepared on the basis of a 1-2/3 mold volume purge.

In most cases, only one set of purging equipment is necessary to synchronize purging with teeming. In selecting a purge flow, the average time of teeming an ingot must be established. This average time is the time to teem a complete heat divided by the number of ingots poured. The actual purge time would be the average teem time less the time for moving the purging equipment from one ingot mold to the next. To illustrate, let us assume that teeming a 100-ton heat into ten 10-ton ingots requires 22½ min. Actual filling of a mold requires less than 2¼ min, but hot-top pouring and moving the ladle accounts for the additional time. Allowing 15 sec for removing and replacing the purge gas distributor, the purge time would then be about 2 min. Thus, for a 10-ton ingot the purge argon flow rate from Fig. 3 should be 2000 cu ft per hr.

When casting small ingots, the average teeming time per ingot may approach the time necessary to remove the purge gas distributor, reseal the aluminum hot-top cover, and place the distributor in the next mold. Obviously, the purge flow compatible with this condition would approach infinity. In this circumstance, the use of two sets of purge equipment would be necessary. This must be determined in the individual shop.

Teem protection

Protecting the teem stream is necessary to eliminate exposing the molten steel to oxidizing conditions from the ladle to the ingot and to maintain low oxygen content in the mold. In normal practice, the falling metal stream not only aspirates air into the mold, but heats mold gases causing them to rise and draw in air. In an early test with an argon purged mold without teem protection, 13 pct O_2 (or 62 pct air) was found in the mold atmosphere when the mold was half filled during teeming.

A physical barrier between the teem protection argon and the surrounding air is necessary to achieve a completely inert atmosphere around the teem stream. Argon jets directed at the stream can not provide adequate protection. A jet of gas is diluted by the ambient gases as the square of the distance from the jet source.

A teem stream protector for distributing argon around the teem stream and providing the mechanical barrier between the argon and air is shown in Fig. 2. The protector is constructed of light-gage steel welded into a cylinder. It is attached to the bottom of the ladle concentric with the nozzle. The top of the protector must surface on the ladle bottom or the nozzle retaining plate. The diameter of the protector should be slightly less than the minimum inside hot top dimension. Sometimes it is necessary to make a stepped cylinder so that its upper diameter can clear the nozzle retaining plate.

The length of the protecting cylinder should be approximately equal to its diameter. This allows a fanning teem stream to clear the bottom and, although small amounts of splash may reach the cylinder, these will not be harmful. This protector length also achieves the greatest visibility without interfering with a fanning stream.

An alternate, but less desirable, position for the teem protector is resting on the mold hot top. The latter position should be used only if some physical condition makes suspension from the ladle impossible. Placing the teem protector on the hot top requires a supporting collar which limits visibility. When moving from mold to mold, the protector must then be moved by hand. Also, it is impractical to lower the ladle to touch the protector. This leaves a space for air infiltration and increases the required teem protection argon flow to maintain an inert atmosphere.

Before teeming begins, the ladle with the suspended teem protector should be centered over the hot top opening with the bottom of the cylinder less than ½ in. from the aluminum foil. The protector aids in locating the nozzle over the center of the mold. About 5 sec before the nozzle is opened, the teem protection argon should be turned on. A flow rate of 1100 cu ft per hr with a 12-in. diam protector prevents mold atmosphere contamination without disturbing the teem stream.

As the molten steel falls from the nozzle a hole is melted through the aluminum foil to approximately the outside diameter of the protector. Expansion of the argon mold atmosphere, argon from the teem protection flow, and gases from the decomposition of the mold coating raise the aluminum foil around the protector forming an effective seal against the back diffusion of air. The temporary seal is maintained until the mold is nearly filled. Finally, the aluminum melts away when the metal reaches the hot top, allowing observation of the metal level.

Results

Argon casting is effective in three general areas of application. First, with steels which are accepted on the basis of cleanliness, argon casting minimizes non-metallic inclusions which form on exposing the molten metal to air. Second, inert-atmosphere pro-

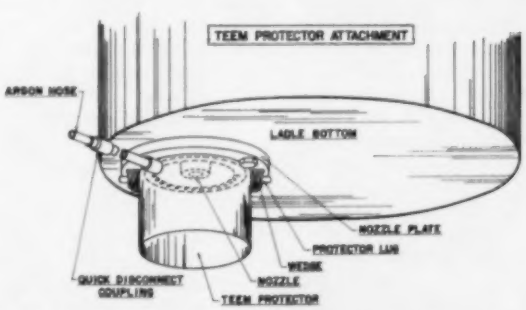


Fig. 2—Teem stream protector for distributing argon around the teem stream and for protection from the atmosphere.

tection reduces conditioning losses by decreasing surface oxidation during teeming. Thirdly, the effects of special vacuum treatments are preserved where steel is vacuum degassed then teemed from the ladle through air. The inert atmosphere eliminates the reaction between molten steel and oxygen, nitrogen, or water vapor which would otherwise partially nullify the effects of vacuum treatment.

Effects on cleanliness

Aircraft quality steels in particular are evaluated on the basis of their relative lack of nonmetallic inclusions. Inclusions originating prior to teeming have a chance to float up into the slag cover—the degree of removal being dependent upon size, relative density, metal temperature, and holding time. However, as previously discussed, oxides formed on exposure to air during the teem have little chance to float out due to rapidly changing conditions which retard their movement.

In the studies of the effects of argon casting, it was soon recognized that evaluation could not be made on a heat-to-heat comparison. Variations contributed randomly by other operations in the melting process affect the number of inclusions remaining in the ingot metal. It was also known that cleanliness varied within the ingots cast from one heat of steel. The first and last ingots poured from a heat are generally less clean than those in the middle of a heat. Therefore, in tests of argon casting, pairs of comparison ingots were cast in the first, middle, and last portions of a heat. In a test pair, one ingot was argon cast and the next adjacent ingot was air cast according to normal practices. In a cooperative test with Atlas Steels Ltd., Welland, Ontario, Canada, approx 150 ingots were cast and examined for comparison purposes. During this test, the argon casting process was still under development. On each argon cast ingot, the mold atmosphere oxygen content was followed to determine the extent of purging and atmosphere quality during the teem. The aim for oxygen content at the end of purging was 0.25 pct or less. Some tests were discarded because gas analysis showed incomplete elimination of air. Faults in equipment and technique were corrected as the experimental program progressed.

The inclusion ratings were established according to techniques used at Atlas Steels, which have been previously reported in the Proceedings of the AIME Electric Furnace Conference by Atlas personnel.[4] All the steels used in the tests were melted according to normal procedures established for aircraft quality steel. The steels and their nominal compositions are listed in Table I. Fig. 3 shows the results of the side-by-side inclusion rating comparison within individual heats. The calculated percentage improvement is the difference between the average of the air cast and the average of the argon cast inclusion ratings divided by the average air cast rating. It will be noted that in one heat the average air cast inclusion rating is better than the average argon cast inclusion rating. This illustrates the variability in ratings from ingot to ingot. In this heat the inclusion ratings for the six test ingots were fairly uniform with one very low rating on an air cast ingot and one quite high rating on its argon cast mate. If these two ratings had been discarded in developing the average rating there would have been a 5 pct improvement favoring argon casting on this heat.

The data from all the heats were studied statistically. It was found with 99 pct confidence that there was an improvement in inclusion ratings with argon casting. Also, with 90 pct confidence the average improvement on inclusion rating with argon casting will fall within the range of 30 to 65 pct. This has been substantiated during a year's production experience at Atlas Steels. In Atlas' quality control procedures strategically-located samples from blooms of six ingots from each scheduled aircraft quality heat are given a preliminary examination to establish an average inclusion rating. This determines whether the heat will be accepted for further inspection to meet the specifications of aircraft-quality steel orders. At the beginning of 1959, the inclusion rating level for heat acceptance was reduced by 40 pct, without an increase in the rejection rate, indicating a step forward in overall quality of product. Other improvements have been incorporated in Atlas' melting practices; however, argon casting has substantially improved quality.

Effects on ingot surface

Application of argon casting to improve ingot surface and reduce conditioning requirements has been evaluated to date on stainless and specialty alloys because it could most easily be justified in these materials. In the Atlas investigation, approx 90 comparison ingots from 15 heats were cast and followed to determine differences in metal removal required during primary conditioning. Twelve of the heats were 430 stainless, one was 405, and two were 316. Ingots were cast in test pairs alternately down the line of ingots. Blooms were weighed before and after primary conditioning following ingot break-down and cropping.

The percentage of metal removed in primary conditioning ranged between 3.5 and 9 pct. From these tests, it appears that argon casting is more effective on austenitic stainless steels. However, the results are not completely conclusive since only two heats of 316 were included in the investigation. Statistically, there was a reduction in grinding losses using argon casting with 90 pct confidence. Assuming a 90 pct confidence limit, the average reduction divided by the normal conditioning losses will lie between 3.5 and 15.5 pct. Considering the processes involved in removing metal during conditioning, argon casting will effectively increase production rate and reduce metal removal required by about 10 pct.

Argon casting has been adopted by Wrought Alloy Dept., Haynes Stellite Co., div. of Union Carbide Corp., Kokomo, Indiana, for its reduction of conditioning losses. To evaluate the effectiveness of argon casting on conditioning a nickel-based superalloy, 38 ingots were cast from 19 heats. Two 2.5-ton ingots were cast per heat from a 5-ton electric furnace. To minimize effects of early or late teeming, argon casting was applied alternately to the first and second ingots of each heat.

Finish grinding loss was the criterion for comparing the effects of argon casting and air casting on the ingot surface. After casting, the ingots are normally spot conditioned to remove gross surface defects. Average losses from this operation were essentially equal on both air and argon cast ingots. After hot top cropping, the ingots were forged to break down cast structure and subsequently ground. Each ingot was weighed before and after grinding to determine weight loss. The finish grinding losses

ranged between 3.5 and 8.7 pct. In this test, the argon ingots had less grinding losses than the air cast ingots. Comparison of grinding losses are shown in Fig. 4. Argon cast ingots averaged 17.5 pct less metal removed in final conditioning. Statistically, the average reduction in grinding losses will fall between 11.2 and 24.4 pct with 90 pct confidence.

Effect on vacuum-treated steels

Currently, more and more steel is being vacuum treated to reduce dissolved gas contents and increase cleanliness. The vacuum-treating processes include vacuum casting, vacuum stream degassing from ladle to ladle, vacuum ladle degassing (A. Finkl & Sons process), the reciprocating vacuum vessel (Dortmund-Hoerder), and the vacuum siphon process. In vacuum casting, the molten steel is protected from atmospheric contamination. However, in the vacuum ladle methods unless an inert atmosphere is provided at teeming, exposure to air may cause oxygen, nitrogen, and hydrogen to be redissolved in the steel.

A test of argon casting applied to vacuum ladle degassed 4340 steel was conducted at A. Finkl & Sons, Chicago, Ill. The results of these tests have been previously reported by Finkl personnel[7]. The test consisted of two heats, each with an argon cast and an air cast ingot. Six samples from each test ingot were analyzed by vacuum fusion for dissolved hydrogen, oxygen and nitrogen. Results are presented in *Argon Teeming of Degassed Steel* which also appears in this issue.

It is apparent from these results that the argon cast ingots were protected against pickup of gas components in air. The relative difference in individual gas contents reflect the reactivity of each gas and its partial pressure in ambient air. The amount of hydrogen pickup is dependent on several factors.

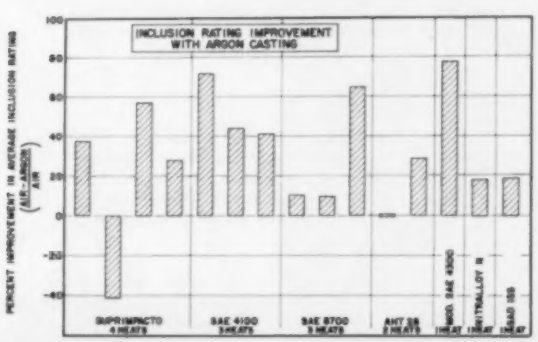
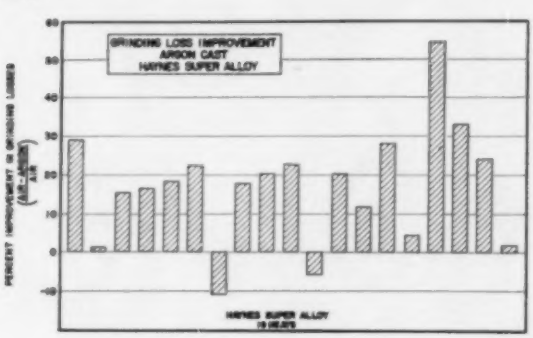


Fig. 3—Above, Effect of argon casting on inclusion rating. Fig. 4—Below, Effect of argon casting on grinding losses for Haynes Superalloy.



Sources of hydrogen include the water vapor contents of the atmosphere and decomposition products of mold coatings. Hydrogen pickup from volatile portions of mold coating would be essentially equal in both the argon and air cast ingots. Therefore, differences in final hydrogen content can be attributed to reaction with water vapor in the surrounding atmosphere. Oxygen and nitrogen differences are essentially equal and are not in proportion to their partial pressures in air. The greater reactivity of oxygen relative to nitrogen is reflected in the increased oxygen pickup.

Economics

The actual cost of utilizing the argon casting process is affected by several factors; these include method of teeming, either direct or using an intermediate tundish; the size of the ingots; and the teeming rate. Mold purging requires 7 cu ft of argon per ingot ton of steel. At a pouring rate of 5 tons per min, argon consumption for teem protection is approx 3.5 cu ft per ingot ton. Total argon consumption (mold purging plus teem protection) has ranged between 9 and 15 cu ft per ingot ton with direct pouring. Argon prices are volume dependent and normally range between $0.05 and $0.10 per cu ft. Using 15 cu ft of argon per ton plus an allowance of $0.05 per ton for mold sealing, the cost of consumables for argon casting will vary between $0.80 and $1.55 per ton. With proper argon flow control apparatus it has been found that no increase in the pit crew is required.

There are appreciable dollar values attached to reduction of inclusion count and conditioning losses resulting from argon casting; however, these can only be determined on an individual mill basis.

Summary

It has been shown that proper argon protected teeming can have selected application in steelmaking processes:

1) While the process requires some care in obtaining a completely inert or protected atmosphere, it is simple and has been developed to the point that it does not require extra manpower;
2) The utilization of an argon protective atmosphere can be economically justified in selected applications;
3) It is substantially effective in reducing nonmetallic inclusions in the final product. It eliminates those which have their source in the last exposure of the liquid metal to air;
4) It is effective in reducing conditioning losses in high-alloy steels, specialty steels, and in certain other alloys; and
5) It maintains the gas contents which have been minimized by vacuum treatment.

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ARGON TEEMING OF DEGASSED STEEL

This article describes the application of argon teeming techniques to degassed steel for the production of high-quality forgings.

by William Wilson

The A. Finkl & Sons Co.—like all producers of heavy forgings—is constantly on the alert for improved processing methods. Our objectives are to increase the useable strength, cleanliness, ductility, notch toughness, and machinability of our steels.

At the 1958 Electric Furnace Conference, C. W. Finkl presented the results of our new combination of helium purging and ladle degassing. Steel treated in this manner exhibits a very tight stream during teeming, but there exists the possibility of gas pick-up. The argon teeming technique developed by the Linde Co. Development Laboratory practically excludes gas pick-up during teeming. This process has two phases: displacement of the air in the ingot mold with argon, and provision of an argon shroud around the stream during its descent into the mold. A number of the original process details have been changed to fit Finkl's specific requirements.

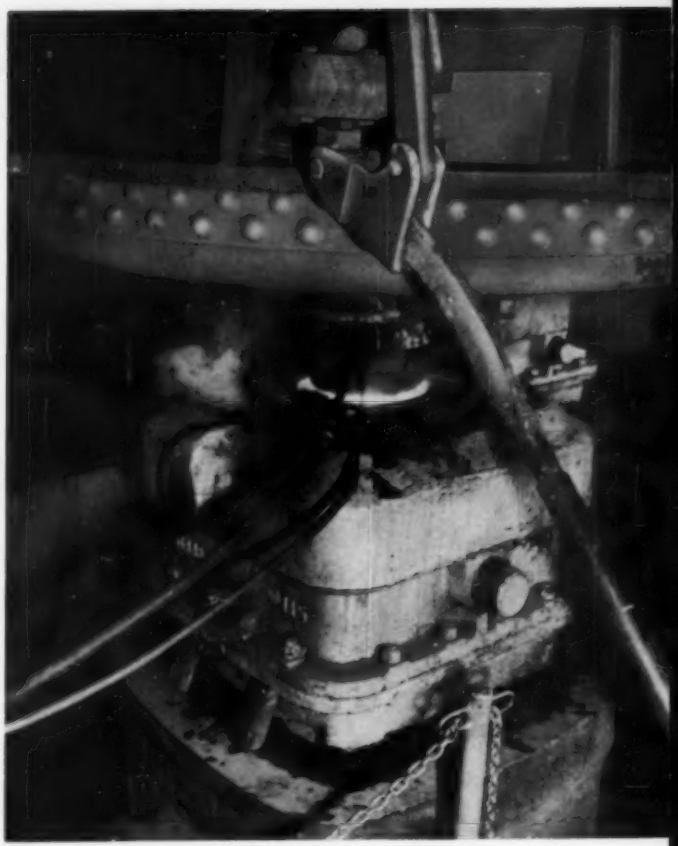
Mechanical details

In preparation for argon teeming, the ingot molds receive their regular cleaning and inspection but are then sealed. The mating surfaces on the stool and mold are gasketed with asbestos paper or fiberboard and silicate cement.

With C&D hot tops, the annular gap between the mold and hot top is packed with asbestos rope and covered with a mastic. The hot top opening is covered with a 0.004-in. aluminum foil disk resting on mastic and held in place with a steel ring. A flap is cut in the foil to permit insertion of the argon

(*Continued on page 352*)

WILLIAM WILSON is with A. Finkl & Sons Co., Chicago. This paper is presented as a discussion to the Hoffman, Bailey, and Holmes paper which is published in this issue of Journal of Metals.

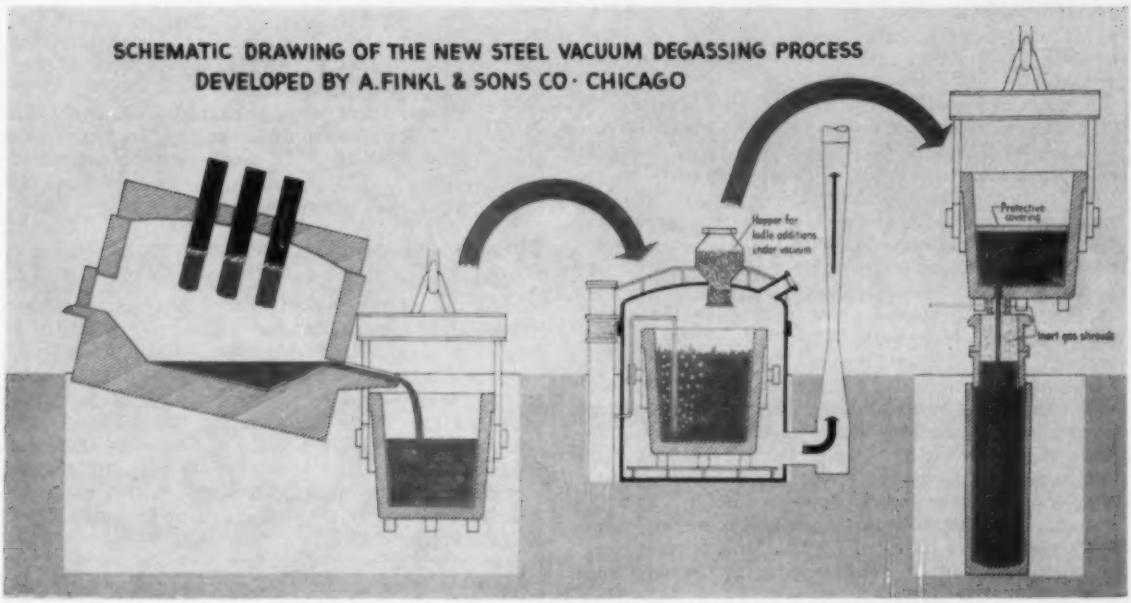


Pouring vacuum-degassed nickel-chrome-molly steel through a blanket of argon into an ingot mold. The larger pipe delivers argon, and the smaller one bleeds a continuous gas sample during teeming.

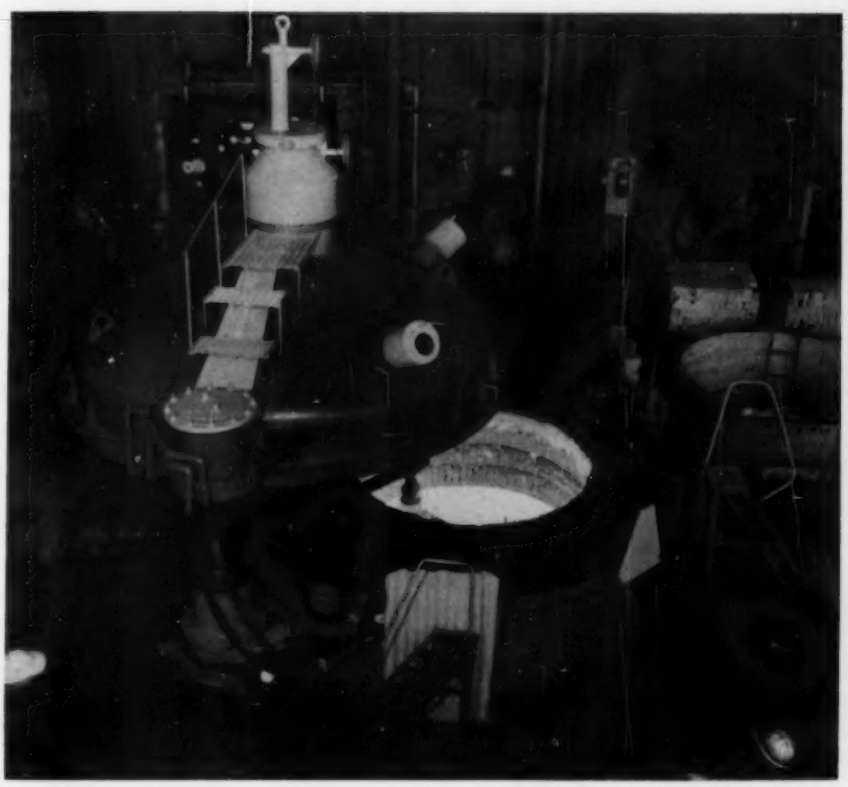
But before pouring, ingot molds are filled with argon gas by a diffuser which is lowered into the bottom of the mold. An oxygen testing device (left) records the atmosphere inside of the mold. The mold is closed prior to casting.

BUT,

Argon Teeming is not the whole story;
The Process is a combination of
VACUUM DEGASSING
FOLLOWED BY
ARGON TEEMING


SCHEMATIC DRAWING OF THE NEW STEEL VACUUM DEGASSING PROCESS
DEVELOPED BY A.FINKL & SONS CO · CHICAGO
Hopper for ladle additions under vacuum
Protective covering
Inert gas shrouds

TAPPING **DEGASSING** **TEEMING**



The Finkl vacuum degassing chamber with the new hopper for additives installed in the top of the chamber. In the latter part of the degassing cycle, various additives, such as dehydrated lime and aluminum, are released to the molten steel in the ladle.

diffuser and a gas sampling tube. After filling, the flap is resealed with tape.

A teem stream protector is attached to the bottom of the ladle. During teeming, this directs 1100 cu ft per hr of gas radially at the stream to form an argon shroud.

Operational details

It is Finkl's practice to argon fill the molds in teeming order, and to complete filling the last mold before teeming the first ingot. This practice is feasible because eight or fewer ingots are teemed per heat.

The air in each mold is displaced by lowering a 5 in. diam x 10 in. diffuser to the bottom and filling with argon. Using 3000 cu ft per hr, a 23-in. ingot mold for an 8000-lb ingot is filled in 30 sec.

A Leeds & Northrup oxygen analyzer—recalibrated for argon with analyzed mixtures—is used to check the residual oxygen. With a sampling tube at the top of the ingot body and the prescribed filling conditions, the residual oxygen is always below 0.5 pct and approaches zero. A well-sealed mold will maintain less than 1 pct of oxygen until teemed.

The argon source and oxygen analyzer are then connected to the teem stream protector. Before each opening of the nozzle a flow of 1800 cu ft per hr of argon is maintained for 10 sec. During teeming, this flow is reduced to 800 cu ft per hr, thereby keeping oxygen in the teem stream protector below 1 pct.

Argon consumption averages 20 cu ft per ingot ton with a range of 18 to 22. This high argon consumption results from the low average teeming rate of 1-1/3 tons per min.

Gas analysis

Finkl's first tests were with 40-in. ingots—two per heat—each weighing 17.5 tons. Two heats were used. On the first heat, the first ingot was air teemed and the second ingot was argon teemed. The order was reversed on the second heat.

Gas samples were cored from the surface, midpoint, and center locations of blocks forged at mid-height of all four ingots. The average gas contents are given below with a probability statement that the difference is due to chance:

Teemed in	No. of Analyses	H_2	ppm O_2	N_2
Argon	12	1.5	31	26
Air	12	1.7	40	37
Probability		0.01	0.2	0.001

These results definitely show a gas pick-up during teeming in air and indicate that it would be of concern in critical applications.

Etched half-slices were prepared from each pair of blocks and etched simultaneously. In Fig. 1 two etched half-slices of AISI 4340 steel are compared. Ingot No. 1 (293170-1) was argon teemed, and the second ingot was air teemed.

Cleanliness

Wet magnetic particle inspection is becoming essential for all super-duty steels. A quarter section immediately above and/or below the product is forged to a standard diameter round. After heat treatment and smooth finishing, the test bar is circularly magnetized, while suspension of magnetic particles are made to flow on the surface. The length of the indication is taken to be the inclusion lengths. Usually, the length represents an aligned group which are linked together by the magnetic powder because single inclusions of fractional inch lengths are a rarity.

Recently 275 sq ft of surface were examined from 11 heats melted to aircraft practice, degassed in the ladle, and teemed with argon. Summarizing this examination, the following frequencies were evaluated:

	Frequency: Magnetic Indications		
Size, in.	1/10 to 1/8	≥⅛ ≤¼	≥¼ ≤½
Frequency per sq ft	4.5	1.5	0.13

The forgings produced from these ingots also met very rigid ultrasonic inspection. Each ingot cast in argon was reported by the forge shop as having good surface.

Mechanical Properties

The above ingots were forged to 15½-in. diam rounds and heat treated at this size. A high-side AISI 4340 with above normal silicon and molybdenum gave the following properties on 70 forgings:

Tensile Strength, psi	0.2 pct Yield Strength, psi	Elongation in 2 in., pct	Reduction of area, pct
Min 190,000	178,000	9.5	28
Max 210,000	195,000	13.5	43

Conclusion

Argon teeming has permitted an evaluation of gas pick-up when teeming degassed steel. Avoiding contamination from this source is essential in critical applications.

Other benefits are improved macro-etch quality, low magnetic particle counts, improved ultrasonic quality, better ductility, and good ingot surface.

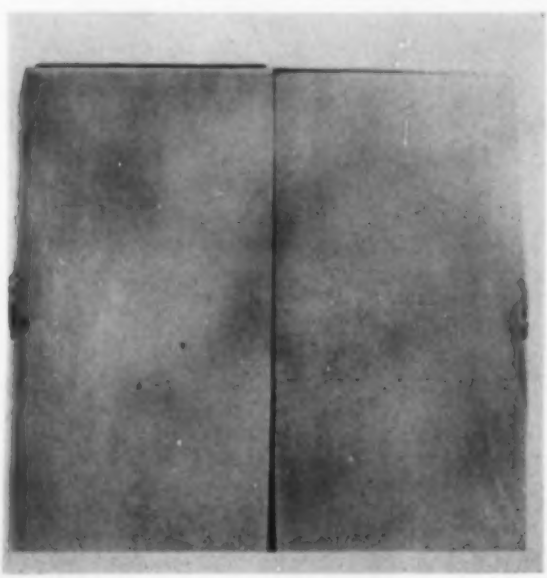


Fig. 1—Etched slices of AISI 4340 steel: at left, argon teemed; and at right, air teemed.

ROLE OF BACTERIA IN THE ALTERATION OF SULFIDE MINERALS

This paper describes studies undertaken with the objective of applying bacterial oxidation reactions more effectively for the recovery of copper from mine waste.

by E. E. Malouf and J. D. Prater

The rapid accumulation of mine waste in the orderly development of the Utah copper mine of the Kennecott Copper Corp. has literally created mountains where canyons used to be. This waste—accumulating at the rate of 200,000 tpd—is in most cases barren or contains only traces of copper minerals. But because of the magnitude of these dumps, these traces add up to a substantial amount of copper. To recover economically some portion of this copper constitutes a real technological challenge.

Several years ago, investigators working at the Brigham Young University, under a Kennecott Copper Corp. grant, discovered the presence of bacteria in Bingham Canyon mine water that are capable of oxidizing pyrite to ferric sulfate and sulfuric acid and oxidizing copper sulfide minerals to copper sulfate. These bacteria play an important role in the waste dump leaching process, and, at the Kennecott Research Center, studies have been in progress for several years with the objective of applying these bacterial oxidation reactions more effectively to the recovery of copper from mine waste.

Two strains of bacteria have been identified in the waters from copper mine waste leaching operations.[1, 2, 3] The first, *thiobacillus thio-oxidans*, is capable of oxidizing reduced forms of sulfur, such as sulfide sulfur, to sulfate ions. The second, *thiobacillus ferro-oxidans*, is capable of oxidizing dissolved ferrous iron to the ferric state. Both strains* are autotrophic, capable of living on inorganic substances—such as dissolved salts, nitrogenous material, carbon dioxide, and oxygen—that are present in natural water. Both strains derive the energy for their life-cycle from the sulfur- and iron-oxidizing reactions noted above.

The laboratory, pilot, and field studies conducted by the Kennecott Research Center have been concerned primarily with determining the conditions under which a bacterial oxidation can occur and the conditions which promote high rates of bacterial activity as well as high rates of leaching copper from mine waste. In the course of these studies, bacterial oxidation has been achieved under conditions far removed from those present in mine waste dumps and has been observed at much higher rates than can be obtained in an uncontrolled waste leaching operation. In fact, the basic requirements for bacterial activity having been determined, bacterial oxidation of ferrous iron, particularly, can be made a sufficiently rapid and economical process to be of general hydrometallurgical interest.

A very effective lixiviant for copper sulfide minerals can be generated by using bacterial oxidation to oxidize pyrite to sulfuric acid and ferrous sulfate, and the same bacterial oxidation to convert rapidly the ferrous iron in solution to ferric iron. The acid obtained in the oxidation of the pyrite is sufficient to hold the ferric sulfate in solution until it comes in contact with the copper sulfide minerals. These minerals are oxidized to produce copper sulfate, and the ferric sulfate is reduced to ferrous sulfate to provide again the food for bacterial oxidation.

The heat generated by the oxidation of the minerals has an accelerating effect on the leaching of copper from mine waste. It has been noted in one

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* Throughout the remainder of the text of this paper the two strains of bacteria—*thiobacillus thio-oxidans* and *thiobacillus ferro-oxidans*—will be referred to, without differentiation, as *bacteria* or *oxidizing bacteria* for the sake of brevity. No strains of bacteria other than these two are referred to in this paper, nor can they be considered to be a part of the subject matter of this paper.

large waste dump where leaching of copper had been started with ferric sulfate solution that sufficient heat was generated to raise the temperature of the water 25°F. Controlled laboratory tests have indicated that this increase in temperature increased the leaching rate three-fold over that obtained with systems at ambient temperatures.

The bacteria that are found in mine waters exist under a fairly wide range of environmental conditions, but, in general, the mine water is weakly acidic and contains low concentrations of various salts. However, in the laboratory, it has been possible to increase the tolerance of bacteria to high acid and salt concentrations in solutions used for leaching waste. Similarly, in the laboratory it has been possible to increase the rate of bacterial oxidation action markedly above that observed in nature. Active bacterial cultures have been established in solutions containing as much as 15 gpl of copper, as much as 40 gpl of iron, and in sulfuric acid concentrations sufficiently high to yield an indicated pH of 0.5. Similarly, in the laboratory, iron oxidation rates have been obtained as high as 20 g of iron converted to the ferric state per liter of solution per 24 hr and pyrite dissolution rates as high as 1.2 pct of the pyrite present in the system per 24 hr. The bacterial activity, under these conditions, is far greater than encountered in nature or in processes employing corresponding chemical oxidation based solely upon air and water.

Fig. 1—Laboratory apparatus for bacterial oxidation studies.

Methods of study

Standard bacteriological methods do not lend themselves to the study of these oxidizing bacteria. So, our studies have been based on following and evaluating the net chemical changes that occur in a controlled system by reason of the bacteria that are present in that system. This approach has considerable practical value, for it generally involves rates of reaction, and these rate data are of major importance in development and evaluation of processes based on bacterial activity. However, general descriptions of the bacteria or direct data on their concentration in a system are not obtained by this technique.

In most instances two chemical criteria are used to evaluate bacterial activity: 1) changes in the pH of the system, and 2) changes in concentration of ferrous iron. In certain cases, changes in total iron, total copper, or total zinc concentrations have been employed to gauge the rate of dissolution of sulfide minerals.

Typical apparatus used in bacterial studies is shown in Fig. 1. This apparatus consists of a column into which can be charged mineral specimens, inert rock, and bacterial cultures, and an airlift by means of which the aqueous bacterial culture can be circulated and aerated. Larger test apparatus has been constructed for certain studies, but the basic principle of using an airlift to provide for aeration of the bacterial solution and for circulation of the solution through a bed of solid particles has been followed.

In practically all studies, a suitable overall rate of chemical change is determined for a particular defined system in which bacteria are present and compared with a corresponding rate in a similar system in which bacterial action has been stopped by addition of a suitable bactericide. The difference in the observed rates is taken as a measure of bacterial activity or bacterial culture strength. Fig. 2 is a typical presentation of data from such a study, presented in the form of rate curves for the bacterial leaching of chalcopyrite. Similar types of data regarding the rate of conversion of ferrous iron to ferric iron have been employed to determine the *strength* of bacterial cultures, to demonstrate the presence of bacteria in various natural rock and mineral samples, and to determine whether or not a culture has been destroyed by a change in, or addition to, an established environment.

Table I. Effect of Surface of Pyrite on Its Rate of Bacterial Dissolution

FeS_2 present, g	Iron dissolved per day, g
10	0.008
25	0.024
50	0.036
75	0.051
100	0.053

Tolerance of bacteria to ions

The oxidizing bacteria can exist under a wide range of concentration of ions in solution, although they are sensitive to sudden and drastic changes in the composition of the solutions which constitute their environment. Tolerance for relatively high concentration of ions, which normally would sterilize a culture, can be developed by gradually increasing the concentration of the ion in solution. A notable example of this was found with the zinc ion. It was found that concentrations of zinc as low as 50 ppm were adequate to sterilize the bacteria occurring in Utah div. mine water, although tests showed that the oxidation of sphalerite and solubilization of the zinc by bacterial oxidation are possible (Fig. 3). Tolerance for zinc ion was obtained by contacting a viable culture with sphalerite, thus effecting a slow increase in zinc ion concentration to which the bacteria became adapted.

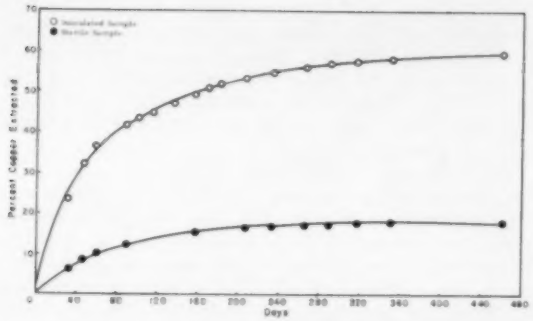
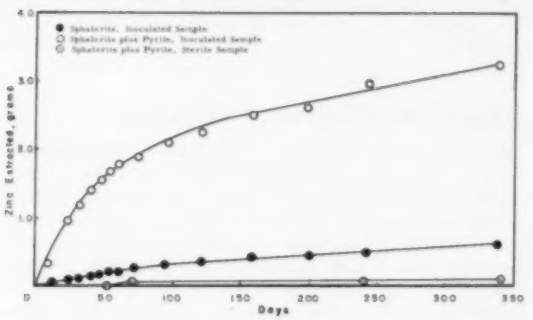


Fig. 2—Above, Comparative rate of copper extraction from chalcopyrite with bacterial and sterile solutions. Fig. 3—Below, of sphalerite and a mixture of sphalerite and pyrite.



Similar adaptation to high metal ion concentrations has been observed in the case of copper which is toxic to the bacteria unless, by adaptation, a tolerance has been developed. By slowly developing the tolerance for copper, it has been possible to produce active bacterial cultures in solutions containing as high as 15 gpl of copper.

Effect of radiation, temperature, and pH

Bacterial cultures become inactive, but are not destroyed, when exposed to direct sunlight. Experiments have been made to evaluate bacterial activity in open ponds with solution depths of up to 2 ft. Virtually no bacterial oxidation was noted in these ponds, even though the solutions were in night-time darkness during 50 pct of the time, and viable bacterial cultures were present. Apparently, in shallow ponds sufficient radiation is transmitted through up to 2 ft of water to inhibit bacterial activity but not to destroy the bacteria present. The bacteria are extremely sensitive to ultra-violet light, for only short time exposure to this radiation will completely sterilize a bacterial culture.

Temperature has been found to exert a very pronounced effect on the rate of bacterial conversion of Fe^{++} to Fe^{+++} with maximum bacterial activity occurring at approximately 35°C. Temperatures above 50°C have been found to destroy the bacteria and to sterilize a culture solution; below 35°C, the rate of bacterial action decreases non-linearly as the temperature is reduced. Fig. 4 illustrates the effect of culture temperature on the rate of bacterial oxidation of ferrous iron.

The oxidizing bacteria are active only in acid media. In general, bacterial action is most pronounced in a media having a pH of between 2.0 and 3.5. Both above and below this pH range the rate of bacterial oxidation decreases, and at pH values above 6.0 bacterial action is almost completely inhibited. In alkaline media (pH 9) the bacteria are destroyed.

Much of the work with these bacteria has been concerned with ferric iron solutions, generated by bacterial action. Control of pH is, in this instance, necessary to prevent precipitation of ferric iron salts from solution. Such pH control does not affect bacterial oxidation rates adversely.

Effect of solid surface

One of the most interesting findings from laboratory studies was that bacterial oxidation rates depend upon the surface of solid material present (as an inert packing) in the oxidation columns. The observations indicated that the bacteria concentrate at the surface of solids in contact with the solution, and build up a large bacterial colony there. Crushed rock particles that are placed in a test column provide the surface for such colonies and, as a result, markedly affect the rate of oxidation that can be achieved in a given system. Quantitative data on the requirement of solid surface area has not been established, but qualitatively it appears that this is one of the more important factors to be considered in development of bacterial oxidation procedures.

Undoubtedly, in the case of the dissolution of sulfide minerals by bacterial oxidation, the specific surface of the sulfide mineral grains influences the rate of dissolution. Only qualitative data and observations regarding this point have been obtained from our laboratory studies. For example, in one series of tests, the total quantity of pyrite of the same sizing analysis was varied in otherwise identical leaching systems. Data from this test series are shown in Table I.

The increase in rate of dissolution of iron from the pyrite is observed to be a non-linear function of the amount of pyrite present, which, under the experimental conditions, was dependent upon the surface of pyrite exposed to the bacterial action. Supporting evidence, indicating the importance of specific surface of sulfide of minerals upon dissolution rate, is the fact that the highest rate of pyrite solubilization achieved in the laboratory was obtained with −325 mesh material.

Effect of solution aeration

The supply of oxygen to cultures of bacteria is, in some respects, the most important factor determining their activity. Supplying oxygen, or air, to a bacterial oxidation system in which solid rock particles are present generally involves two factors: 1) aeration of a portion of the bacterial solution, and 2) circulation of aerated solutions to the site of bacterial activity. These two factors mutually determine the influence of aeration on bacterial activity, and both must be considered in evaluating the performance of a bacterial oxidation system. The importance of solution circulation through beds of crushed rock or other supporting media was not always fully appreciated and, in some early studies, efforts to increase bacterial activity by increasing aeration were negated by failure to provide sufficiently rapid circulation of the aerated solutions through the system.

The relationship between bacterial oxidation rate, degree of aeration of solution, and the rate of circulation of aerated solution through a porous media supporting a bacterial culture is illustrated by the test data presented in Table II. In this test, a total of 11.5 liters of acidic iron solution was circulated con-

Table II. Effect of Aeration Rate and Solution Circulation Rate on the Rate of Bacterial Oxidation of Ferrous Iron (Total Volume of Solution, 11.5 liters)

Aeration Rate, cc/min	Solution Circulation Rate, cc/min	Iron Oxidation Rate, g/24 hr	Iron Oxidation Rate, g/l/24 hr	Utilization of Oxygen, pct
45	750	36.8	3.20	27.16
118	750	45.8	3.98	13.55
125	1500	70.4	6.12	21.00
550	1500	80.0	6.96	4.84

tinuously, but at varying rates, first through a vessel equipped for efficient aeration of the solution, and then a column packed with crushed stone and containing an established bacterial culture. The arrangement thus permitted independent variation of both circulation and aeration rates.

The data show that the iron oxidation rate is increased by increasing the aeration rate or the solution circulation rate. However, the efficiency with which the oxygen content of the aerating gas stream is utilized in the oxidation reactions is markedly dependent upon a proper balance between the rate of aeration and the rate of circulation of the bacterial solutions. Extension of the experiments from which the data in Table II were taken, indicated that, under certain conditions, iron oxidation rates of up to 20 g of iron per liter of solution per 24 hr could be attained; however, in these tests, accurate measurement of the actual aeration and circulation rates were not possible. For comparison, the chemical oxidation of Fe^{++} to Fe^{+++} by aeration, under similar conditions except for the absence of bacteria, proceeds at a rate of approx 0.02 g Fe^{++} oxidized per liter per day—approx one one-thousandth as fast as the bacterial oxidation rate.

Bacterial oxidation of pyrite to sulfuric acid and ferric sulfate also has been extensively studied. As with the bacterial oxidation of ferrous to ferric iron, the rate of bacterial action is dependent upon the rate of aeration and circulation of the bacterial solution. The highest rate of conversion of pyrite to ferric sulfate and sulfuric acid obtained in the laboratory was achieved using 1500 g of −325 mesh pyrite suspended on a crushed quartzite bed and 8 liters of a strong bacterial culture. Over a 7-day period 105.7 g of pyrite were dissolved, equivalent to a dissolution rate of 1.18 pct of the pyrite present in the system per day.

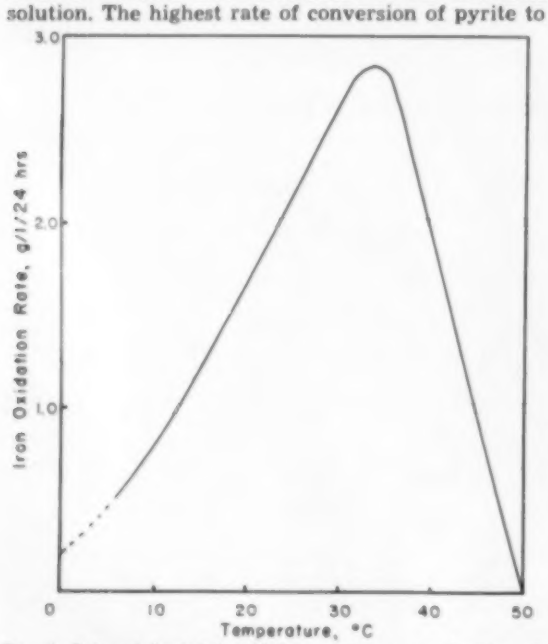


Fig. 4—Rate of bacterial oxidation of iron as a function of temperature.

The efficiency with which the oxygen content of air can be utilized in the bacterial dissolution has been determined only for a system employing coarse pyrite and moderate dissolution rates. This test indicated that the oxidation reactions consumed 4.7 pct of the oxygen present in the volume of air introduced for aeration and circulation of solutions. Projection of the test data indicated that approx 2.4 million cu ft of air at 2.5 psig would be required to oxidize completely and to take into solution 1 ton of pyrite, or, equivalently, approx 1.7 million cu ft of air would be required to produce 1 ton of ferric sulfate plus sulfuric acid from pyrite by bacterial oxidation.

Utilization of bacterial oxidation

As was pointed out earlier, the principal objective of the study of bacterial oxidation processes conducted at the Kennecott Research Center was to determine methods of leaching mine waste dumps more efficiently. These studies have shown that bacterial action can affect the relatively rapid dissolution of sulfide copper minerals and can effectively generate ferric sulfate solutions. Shielding from light, the presence of solid surfaces for bacteria cultures, thorough aeration of bacterial solutions, and high rates of solution circulation have been established as the important controllable process variables. Currently, large-scale dump leaching tests are in progress to evaluate the effectiveness of the control of variables indicated by the laboratory tests. One of these tests involves leaching an isolated dump of freshly-mined waste with ferric sulfate solutions generated by bacterial oxidation.

The bacterial oxidation of ferrous iron solutions may find diverse applications in the hydrometallurgical field, particularly for regeneration of ferric iron solutions in a cyclic leaching process. This conversion can be effected rapidly; it should also be economical, since the principal costs involved are for the sulfuric acid which may be required to maintain the desired ferric iron concentration and for the power consumed in aerating solutions with low-pressure air.

The process of going from ferrous to ferric iron by bacterial oxidation is an inexpensive process for the utilization of gaseous oxygen to form ferric iron. The subsequent reduction of ferric iron to the ferrous state can be considered equivalent to the direct reduction of gaseous oxygen at an electrode. Thus, an economical large-scale fuel cell might possibly be established using the $Fe^{+++} \rightarrow Fe^{++}$ reduction at one electrode and bacterial oxidation, consuming oxygen, to regenerate the ferric iron that has been discharged. This is one of the interesting potential usages—admittedly speculative at this time—for the oxidizing bacteria that occur naturally at the surface of sulfide mineral deposits.

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EVALUATION OF BERYLLIDES

Unique combinations of low density, high strength properties have made beryllium and its alloys extremely important in space applications. This paper presents an evaluation of beryllium intermetallic compounds.

by J. R. Lewis

Interest in beryllium alloys in general has stemmed from the unique combination of low density, high elastic modulus and moderately high melting point of beryllium. For nuclear applications the element is further attractive because of its low absorption cross section to thermal neutrons and fairly good moderating properties. The melting point of beryllium, although moderately high, 2400°F, is not high enough that the element or solid solution alloys of the element can be considered for high temperature applications. The oxidation resistance of beryllium is also not particularly good above 1000°F so that a further limitation is imposed if the element is to be used in oxidizing atmospheres. Since intermetallic compounds often have melting points higher than those of their constituent elements, it is logical to look in this direction for a possible means of extending the useful operating temperature range of beryllium.

The first known study of beryllium intermetallic compounds was reported by Oesterheld[1] in 1916; in 1935, Misch[2] undertook a study of the crystallographic structure of beryllides of copper, nickel, and iron. Work in beryllides was sporadic thereafter until the late 1940's when papers began to appear[3,4] more frequently. The first systematic attempt in this country to survey known and reported beryllides was conducted by Brush Beryllium Company under a USAF contract.[5] The primary intent of this work was not to undertake a study of beryllides per se, but to explore a number of intermetallic systems for potential high temperature oxidation resistant structural materials. Exploratory tests were also made to evaluate oxidation resistance at 2300°F. On the basis of this study, thirteen intermetallic compounds were selected as worthy of further study. Nine of these were beryllides. More recently, data have been published on some mechanical properties by Stonehouse, Paine, and Beaver,[6] and by Booker, Paine and Stonehouse.[7] There are indications that beryllide research and development programs are actively under way in Russia. Several papers in the Russian literature[8-11] have appeared during the past 5 years, and there are persistent rumors of the use of beryllium alloys for high-temperature devices.[12] At least one disclosure was made of beryllide research and development in the United Kingdom when Bissett[13] reported results on $UThBe_{13}$ as a fuel element material under investigation by the Australian Atomic Energy Commission.

Although a relatively small amount of effort has been expended on beryllides compared to other high-temperature materials such as refractory metals and oxides a number of general statements can be made regarding their properties:

1) Densities of beryllides are low, compared to any of the refractory metals and many ceramics. Typical densities are 2.72 g per cc for $ZrBe_{13}$ and 2.92 g per cc for $CbBe_{12}$. For purposes of comparison the density of columbium is 8.57 g per cc; that of BeO is 3.03 g per cc; that of Al_2O_3 is 3.97 g per cc;
2) Oxidation resistance of some beryllides (such as $ZrBe_{13}$, $CbBe_{12}$, $TaBe_{12}$, etc.) is quite good at temperatures as high as 2500°F (comparable to BeO);
3) Mechanical strength, as measured in bending, is higher than that of most ceramics at elevated temperatures;
4) All known beryllides are brittle at room temperature. Some, such as Cb_2Be_{17} and $CbBe_{12}$, deform plastically under load above 2300°F; others have never been observed to exhibit any useful ductility;
5) Thermal shock resistance of beryllides is marginal although this might be improved by control of impurities or by proper design;
6) Hardness of beryllides is of the order of 1000 DPH; and
7) The beryllides must be considered at toxic materials in the same sense as any beryllium compound or alloy.

Properties of beryllides

Melting Points: Since the primary interest in the beryllides has been for their possible use at high temperatures, only those alloys with melting points above 3000°F will be considered in this paper. A number of beryllides are known to exist which fulfill this condition and are listed in Table I. In addition, there is some indication[5,7] that the compounds Hf_2Be_{17}, WBe_{21}, WBe_{12}, and $CeBe_{13}$ may have melting points above 3000°F.

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Table I. Beryllides Melting Above 3000°F

Beryllide	Melting Point °F	Melting Point °C	Reference
$CrBe_2$	3340	1840	13
$LaBe_{13}$	>3150	>1720	5
$MoBe_2$	3340	1840	5
VBe_{12} (VBe_{12} ?)	~3100	~1700	6, 16
$ZrBe_{13}$	3500	1930	6
Zr_2Be_{17}	~3500	~1930	6
$MoBe_{12}$	~3100	~1700	6
$CbBe_{12}$	3100	1700	6
Cb_2Be_{17}	>3100	>1700	6
$TaBe_{13}$	3360	1850	6
Ta_2Be_{17}	3610	1980	6

It will be noted that in several cases, the intermetallic compound offers no advantage in melting point over the *metallic* constituent. In fact, the only cases where the melting point is clearly higher than that of both beryllium and the second element are $LaBe_{13}$, $ZrBe_{13}$ and Zr_2Be_{17}.

Density and Crystallography: Densities of the higher-melting beryllides are tabulated in Table II. Crystallographic structures are also noted. The crystal structures are complex, with large numbers of atoms per unit cell and high coordination numbers. With the exception of the MBe_{12} type, no predictions have been advanced as to the mechanism by which dislocations might move in these structures to provide plastic slip deformation. It does not seem probable that many of the beryllides in Table II will exhibit ductile behavior—at least at temperatures where plastic behavior is other than diffusion controlled.

Fabrication

Because of the relatively high vapor pressure of beryllium at elevated temperatures, and because of the necessity for close control of composition if one is to obtain a single phase intermetallic compound of specific type, it is advisable to produce the beryllides at as low a temperature as possible. For these reasons, melting operations are generally avoided, and the alloys are produced by cold-pressing and sintering or by hot-pressing techniques.[5,6,23] Hot-pressing seems to be generally favored in spite of the risk of reaction between the graphite dies and the beryllide; the reason for the preference lies in the shorter time required and in the higher density produced compared to cold-press and sinter techniques. It has been found advisable in some cases to pre-alloy the elemental powders prior to hot-pressing.[23] The pre-alloying technique yields a more homogeneous product, and reportedly some impurities such as aluminum and silicon can be removed in part by screening the pre-reacted powders prior to hot pressing. A general sequence for fabrication of beryllides such as $CbBe_{12}$ and $ZrBe_{13}$ is outlined in Table III.

Densities of beryllide samples produced by hot-pressing have ranged from 92 pct to 100 pct of theoretical density.[6] Compacts prepared via cold-pressing and sintering were of somewhat lower density,[6] but this could be due to the particular compacting and sintering conditions which were used.

The level of individual impurities in typical hot-pressed samples of beryllide was generally below 100 ppm for most impurities.[23] The important exceptions were BeO, which typically was noted to range from 0.8 to 2.0 pct;[6] iron, which usually lay between 500 to 1500 ppm; and silicon of the order of 750 ppm. Aluminum was also present in as much as 0.5 pct in some samples—supposedly from alumina grinding balls or sintering crucibles.

Table II. Density and Crystallographic Structure of Beryllides

Beryllide	Density, g/cc	Crystal Structure	Atoms Per Unit Cell	Reference
$CrBe_2$	4.34	D^4_{6h}; hex.	12	3, 20
$LaBe_{13}$	2.87	O^6_h; fcc	112	3
$MoBe_2$	6.12	D^4_{6h}; hex.	12	5, 20
VBe_{12}	2.37	D^{17}_{4h}; tetrag.	26	8, 11, 16
$ZrBe_{13}$	2.72	O^6_h; fcc	112	5, 18
Zr_2Be_{17}	3.08	$R\bar{3}m$; rhomb.	19	5, 17
$MoBe_{12}$	3.02	D^{17}_{4h}; tetrag.	26	5, 8, 16
$CbBe_{12}$	2.88	D^{17}_{4h}; tetrag.	26	8, 11, 16
Cb_2Be_{17}	3.28	$R\bar{3}m$; rhomb.	19	5, 19
$TaBe_{13}$	4.18	D^{17}_{4h}; tetrag.	26	5, 10
Ta_2Be_{17}	—	$R\bar{3}m$; rhomb.(?)	19(?)	5

The Australian AEC has been conducting work on $UThBe_{13}$ as a dispersed fuel material in high-burnup fuel elements.[23] The beryllide is produced by hot-pressing the metal powders in a graphite die at 2732°F. These compacts are subsequently converted to powder which is then dispersed and clad in beryllium metal. Densities are reportedly better than 94 pct of theoretical.

Most intermetallic compounds, including beryllides, are considered to be brittle materials. In fact, one of the common difficulties of fabrication lies in the tendency of beryllides to fracture during cooling after hot-pressing.[23] Such fractures typically occur in beryllides below or near red-heat (approx 1200°F). At higher temperatures, it may be possible to observe plastic behavior, and under the near-isostatic conditions of extrusion, some beryllides can be worked. The author has extruded $ZrBe_{13}$ with some degree of success at 2750°F. It was possible to extrude from approx 2 in. billet diameter to a rod ¾-in. diam after canning the billet in a heavy (¾ inch wall) molybdenum jacket. The extruded rod was cracked into several sections ranging from 1 to 4 in. in length. From the nature of the cracks, it is probable that they originated at inclusions of Be_2C during cooling of the extruded rod when the differences in thermal contraction between can and core placed the core under tension. Similar attempts to extrude $CbBe_{12}$ at a lower temperature (2200°F) and in a mild steel can were unsuccessful. Failure to obtain acceptable extrusions in this case was attributed to the choice of canning material.

Only one known attempt to melt beryllides has been reported in the open literature. Chubb and Dickerson [24] made a few exploratory arc melts under helium without marked success.

Oxidation resistance

Upon exposure to air at elevated temperatures, a surface film of BeO is formed on beryllide intermetallic compounds. The thickness of the film and its protective character depend upon the composition of the underlying alloy and possibly upon the water vapor content of the air.

A number of beryllides have been found to withstand a temperature of 2300°F for 100 hr with less than 0.001 in. surface oxide penetration.[5] Among these $TaBe_{13}$, $TiBe_{12}$, $TiBe_{13}$, $MoBe_{12}$, Zr_2Be_{17}, $ZrBe_{13}$, $CrBe_2$, $CbBe_{12}$, and Cb_2Be_{17}. Some beryllides, particu-

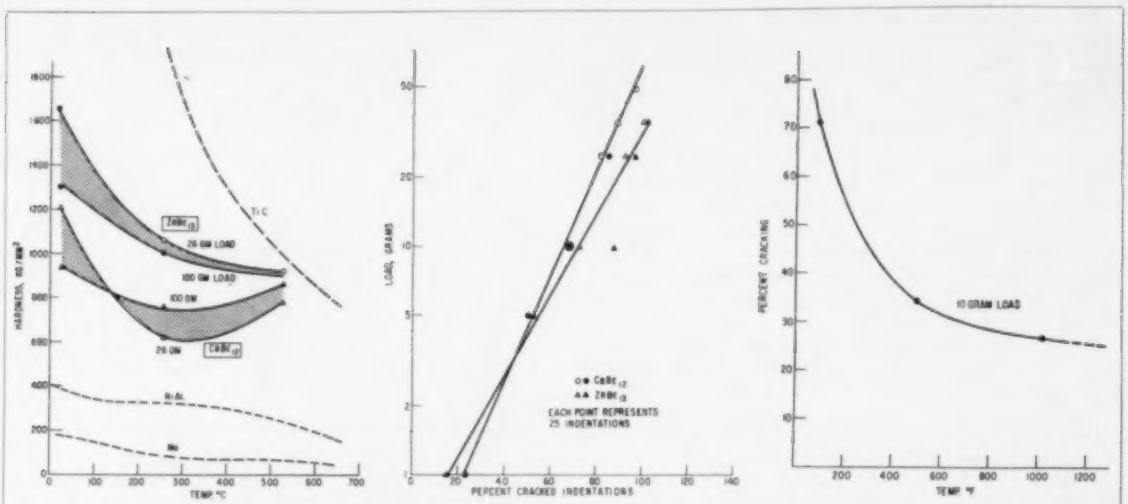

Fig. 1—Left, Microhardness vs temperature for two beryllides and several high-temperature materials.
Fig. 2—Center, Effect of load on cracking microhardness indentations for $CbBe_{12}$ and $ZrBe_{13}$.
Fig. 3—Right, Effect of temperature on crack frequency in microhardness indentations $ZrBe_{13}$.

larly $ZrBe_{13}$, $CbBe_{12}$ and $TaBe_{12}$ have shown promise for operation at 2600°F.[21] The oxide film formed on the beryllides is, at least in the case of MBe_{13} compounds, separated from the underlying body by a substrate of composition M_2Be_{17}.[21] In general, the BeO film is fairly adherent at elevated temperatures. It is inclined to be flakey, however, upon cooling after extended high-temperature exposure. Aitken et al.[25] assign the cause of spalling to the difference in thermal expansion between oxide and substrate. Oxidation rates for a number of beryllides have been found to follow a parabolic or logarithmic curve with respect to time, as would be expected with a protective type of oxide film.

The fact that the protective oxide film on beryllides is a quite stable, high-melting-point oxide is a point in favor of these alloys. Beryllium oxide has a major shortcoming, however, in its vulnerability to attack by water vapor. The reaction,

$$BeO + H_2O \rightarrow BeO \cdot H_2O$$

becomes, for example, quite a serious problem above 2280°F[14,15,28] if the dew point of the surrounding air becomes high. Although exact data are lacking on this point, there is no reason to believe that the BeO film over a beryllide will behave in a manner different than massive BeO. Water vapor attack should be considered a problem with beryllides unless proved otherwise.

Hardness and elastic properties

Because of the brittle behavior of beryllides, it is difficult to obtain a true measurement of hardness by the usual indentation techniques. Even with loads as low as 25 g on a pyramidal diamond indentor, about 90 pct of the indentations will develop cracks in specimens of $CbBe_{13}$ or $ZrBe_{13}$. Because of the effect of cracks in lowering the apparent hardness, any hardness values should be regarded with suspicion. The author has made measurements of hardness of $ZrBe_{13}$ and $CbBe_{12}$ at temperatures up to 975°F. The data of Fig. 1 were collected using 26 g and 100 g loads and measuring only those indentations which were *least cracked*. The two beryllides concerned could be said to be extremely hard, ranging in hardness from about 1000 to 1600 kg per sq mm at room temperature. A minimum hardness of $CbBe_{12}$ at about 550°F is unexplained. Chubb and Dickerson[24] reported room temperature Knoop hardness values of 1100 kg per sq mm for $CbBe_{12}$, 870 for YBe_{13}, and 790 for $ZrBe_{12}$ (?). Alder[29] reports values of about 1300 kg per sq mm for UBe_{13} and $ThBe_{13}$.

If a series of microhardness impressions are made in a brittle material at a given load and temperature and at a given loading rate, it will be found that the fraction of the impressions which develop cracks is for some materials a reproducible number. This fact was noted and reported by Westbrook[30] in the case of quartz, and earlier was suggested by Winchell[31] as a possible means of indexing the brittleness of hard materials. Tests performed on $ZrBe_{13}$ and $CbBe_{12}$ revealed that the fraction of hardness indentations which cracked are for these materials, also, a function of the load on the indentor. Fig. 2 shows the crack frequency versus load results at room temperature. It will be noted that even at loads as small as 1 g, the fraction of cracked indentations is around 20 pct. As the temperature is increased, the fraction of cracked indentations in general decreases, as shown in Fig. 3.

In order to orient these materials in general hardness spectrum, room temperature values for a number of different materials are tabulated in Table IV. The beryllide $ZrBe_{13}$ is one of the hardest intermetallic compounds for which data have been reported.

Table III. Fabrication Outline for Beryllides

Step	Operation	Temperature, °F	Pressure, psi
1	Blend elemental powders	Room Temperature	—
2	Press powder into compacts	Room Temperature	11,000
3	Sinter (Use BeO crucible)	1800-2400 in vacuum	—
4	Crush and mill to −200 mesh powder	Room Temperature	—
5	Hot-press (Graphite dies)	2350-3000	Up to 2500

The only known measurements of Young's Modulus have been performed at Brush Beryllium Co.[5,6,7] Representative values for several beryllides are tabulated in Table V. The values quoted are averages except in cases where widely different figures were encountered in separate references.

The tabulated data were obtained from bend-test results using a three-point loading system. In the case of $CbBe_{12}$, there is evidence[21] that anomalies exist in the elastic modulus over the temperature range 1470 to 2000°F. The evidence is indirect and is based upon changes in the resonance frequency. There are no known data on elastic moduli of beryllides below 2300°F.

No reliable data are available for Poisson's ratio or for the shear modulus of beryllides. In the absence of experimental figures, a reasonable estimate for Poisson's ratio is 0.3 up to say 2000°F, with a gradual increase to the theoretical limit of 0.5 at the onset of plastic behavior.[22]

Modulus of rupture

Most short-time data on the strength of beryllides have been obtained via modulus of rupture tests in bending. This is indirectly a reflection of the brittle nature of the beryllides and their sensitivity to eccentric loads in uniaxial tensile tests. The bulk of recorded testing has been performed at Brush Beryllium Co. by Beaver, Stonehouse, Paine, and Booker.[5,6,7] Representative values are presented in Table VI. Proportional limits (0.2 pct offset) for some of the beryllides are also reported. Stonehouse et al.[5] have reported that at temperatures of 2500°F and 2750°F the beryllides $CbBe_{12}$, Cb_2Be_{17}, "$CbBe_{11}$," and Ta_2Be_{17} have exhibited appreciable plastic deformation in bending. Deflections of as much as 0.25 in. over a 2½ in. span have been noted for specimens of 0.25 in. thickness. At lower temperatures (i.e., at 2300°F) some of the specimens did not yield sufficiently for a 0.2 pct offset yield strength to be calculated. If one examines the data for modulus of rupture of all beryllides which have been reported, it will be noted that there is a range of roughly one order of magnitude between the extreme values of this entire class of materials at a given temperature. It is also interesting to note, on the other hand, that even for a given type of beryllide (e.g., $CbBe_{12}$) the ratio of maximum to minimum values at a given temperature may be as high as 4:1 depending on the grain size. The importance of grain size is well known in dealing with mechanical properties of materials. Kingery[23] has pointed out the marked reduction in modulus of rupture of BeO as the grain size is increased, and Grala[24] noted that coarse-grained specimens of the intermetallic compound NiAl possessed lower tensile strength than did fine grained material. For the beryllides, as reported by Stonehouse et al., the relationship between grain size and rupture modulus would seem to follow the usual pattern.

The effect of impurities upon mechanical strength of beryllides is difficult to assess. In most cases the impurity levels are extremely high, not reported, or are unknown, although Stonehouse et al.[5] claim some correlation between rupture modulus and the presence of aluminum. Certainly one might expect impurities to play an important role in the behavior of these materials.

Rupture modulus values below 2300°F are rather scant for the beryllides. The author has determined values for $CbBe_{12}$ and $ZrBe_{13}$ at room temperature (25,100 and 26,100, respectively) and at −320°F (22,800 and 24,000, respectively), but intermediate data are lacking. Grain size of specimens for the low-temperature tests was approx 20 μ for $CbBe_{12}$ and 30 μ for the $ZrBe_{13}$.

Table IV. Room Temperature Microhardness of Beryllides Compared to Some Other Materials

Material	Hardness, kg/mm²(DPH)	Reference
Al	40	28
W	400	28
AISI 1090 Steel (Oil quench and stress relief)	700 (Rc 60)	29
ZnSe	140	28
$TaSi_2$	1563	28
TiC	3200	27
BeO	1300	30
SiO_2	950	27
$CbAl_3$	460	5
NiAl	330	27
$CrBe_2$	1288	27
Cb_2Be_{17}	1000	5
$CbBe_{12}$	1200	This paper
$MoBe_{12}$	950	5
Ta_2Be_{17}	1120	5
$ZrBe_{13}$	1650	This paper
UBe_{13}	1300	22

Thermal properties

Data on thermal expansion of two beryllides are reproduced in Fig. 4. The values are typically metallic although zirconium beryllide shows a rather large change of slope of α with temperature below 1000°F. An inflection will be noted in the $CbBe_{12}$ curve at about 400°F which corresponds to the hardness minimum noted previously. These curves were plotted from data tabulated by Paine et al.[5] It must be added that Paine does not show the inflection in other curves published in the same reference.

Aitken[25] has given the thermal conductivity of $ZrBe_{13}$ as 19.8 Btu per hr per ft per °F at 2523°F. Brush Beryllium Co. is in the process of measuring thermal conductivity of several intermetallic materials,[6] and has reported values ranging from 16.3 to 20.5 Btu per hr per ft per °F for $NbBe_{12}$ between 1400 to 2500°F.

Impact resistance

Only two references to impact resistance of beryllides have been uncovered; Aitken[25] gives a figure of 12 in.-lb for $ZrBe_{13}$ and 24 in.-lb for $CbBe_{12}$ at 2000°F. These values are two orders of magnitude higher than those reported by Paine,[5] and approximate the values reported by Saller[26] for extruded beryllium metal. The author recently attempted to

Table V. Young's Modulus for Some Beryllides

Material	Temperature, °F	E x 10⁶ psi
Ta_2Be_{17}	2300	17
	2500	13
	2750	10
HF_2Be_{21}	2300	17
	2500	5
$TaBe_{12}$	2300	24
	2500	14
	2750	10
Cb_2Be_{17}	2300	22
	2500	14
$ZrBe_{13}$	2300	20-40
	2500	20
	2750	10
Zr_2Be_{17}	2300	26
	2500	15-30
	2750	10
$MoBe_{12}$	2300	15
	2500	12
	2750	1
$CbBe_{12}$	2300	30
	2500	21
	2750	20

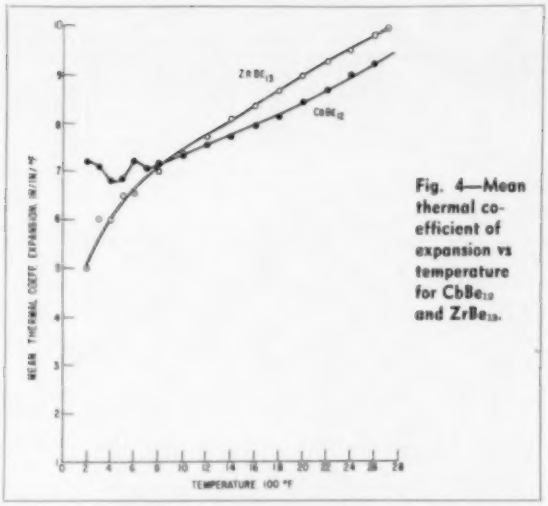

Fig. 4—Mean thermal coefficient of expansion vs temperature for $CbBe_{12}$ and $ZrBe_{13}$.

measure impact toughness of beryllides using a modified Charpy test developed by Soxman et al.[20] This test consists essentially of dropping a known weight through a distance just sufficient to rupture a specimen of standard dimensions. The test specimen is heated to the desired temperature by clasping it between two bus bars and passing an electric current through it. It was not possible to conduct significant high-temperature impact tests with the intended equipment, since the beryllide specimens cracked or spalled under the thermal gradients imposed by heating between two bus bars. Using specimens 1½ in. long x 3/16 in. square, room-temperature impact values of less than 1 in.-lb were obtained for $ZrBe_{13}$ and $CbBe_{12}$. Using similar procedures, Paine[5] reported values of 0.1 in.-lb. This is of the same order of magnitude as has been reported for NiAl,[87] K-152B cermet[88] TiAl,[84] and American hotel china.[89]

Table VI. Mechanical Properties of Some Beryllides

Material	No. Tests	Temp, °F	Grain Size, μ	Modulus Rupture, 10^3 psi	Proportional Limit, 10^3 psi
$CbBe_{12}$	2	2300	50	10.0	—
$CbBe_{12}$	3	2300	11-15	37.4	—
	4	2500	11-15	36.2	—
		2750	11-15	20.3	—
Cb_2Be_{17}	1	2300	9	39.0	9.4
		2500	9	37.5	3.5
		2750	9	16.4	—
$TaBe_{12}$	2	2500	12	53.2	18.5
	2	2500	12	36.0	3.5
	2	2750	12	26.5	3.6
$TaBe_{12}$	2	2700	45	6.5	—
$ZrBe_{13}$	3	2300	25-30	28.2	2.0
	4	2500	25-30	28.1	4.7
	3	2750	25-30	17.9	—
Zr_2Be_{12}	1	2300	80	17.2	—
	1	2500	50	7.5	—
Zr_2Be_{17}	2	2300	30	39.5	23.3
	2	2500	30	27.0	10.2
	1	2750	30	24.4	3.7
$TiBe_{12}$	2	2300	9	11.2	2.0
		2500	9	3.5	—
		2750	9	2.7	—

[a] Data are average values from original data of reference.[5] Data recently published in part II of reference[5] are appreciably lower for $ZrBe_{13}$.

Fractures of impact test specimens of $CbBe_{12}$ and $ZrBe_{13}$ are conchoidal in appearance. Electron photomicrographs revealed occasional evidence of cleavage, but in general the fracture surface was smooth and rounded in appearance at 10,000 X magnification. Inclusions of a second phase were noted in the form of isolated rounded particles of the order of 2 μ diam in the $ZrBe_{13}$ specimens. Fracture surfaces of $CbBe_{12}$, on the other hand, exhibited inclusions as finer particles in patches. The inclusions were not identified.

X-ray diffraction and optical microscopic studies of the impact specimens revealed the zirconium beryllide to consist primarily of $ZrBe_{13}$ with a minor amount of a second phase (Zr_2Be_{17}). The $CbBe_{12}$ specimens were essentially single phase.

Thermal stress resistance

Although claims are occasionally made regarding the good thermal stress resistance of beryllides, these pronouncements do not agree with certain observations: 1) Paine et al.[5] have reported trouble with spalling and rupture of beryllides during or after hot-pressing; 2) Alder[22] reports the resistance to thermal shock of $UThBe_{13}$ to be rather low, with fracture frequently occurring during cooling after hot-pressing; and 3) As reported in this paper, impact specimens commonly crack during heating.

Thermal stresses arise as a result of differential expansion tendencies of different parts of a structure. An excellent review of the factors which influence thermal stresses has been published by Kingery[90]. As pointed out by Buessen,[40] practical experience has shown thermal shock tests do not lead to generally useful data. This is so because resistance to thermal stress is not an intrinsic material property, but is rather a function of five intrinsic properties plus such items as heat-transfer coeffcient, shape, and size, which are external to the material in question. The five intrinsic properties are elastic modulus, tensile strength, thermal coefficient of expansion, Poisson's ratio, and thermal conductivity. Under certain conditions, density, specific heat, and emissivity may also be important.

It is possible to index the thermal stress resistance of a material in terms of one or more factors, depending upon operating conditions. For unsteady state conditions, for example:

$$(1)\quad R = \frac{S\,(1-\mu)}{E\alpha}$$ (Under conditions of high heat-transfer rate.)

$$(2)\quad R' = \frac{S\,(1-\mu)\,k}{E\alpha}$$ (Under conditions of low heat-transfer rate.)

where: R (or R') = stress-resistance index for material in question; α = coefficient of thermal expansion; E = elastic modulus; S = tensile strength; μ = Poisson's ratio; and k = thermal conductivity.

It should be emphasized also that the foregoing relationships apply exactly only to brittle homogeneous isotropic bodies whose physical properties are not sharply temperature dependent.

Using data which appear elsewhere in this report, and assuming a value of 0.3 for Poisson's ratio, the thermal stress resistance factors R and R' have been computed for three typical beryllides and compared

to values tabulated by Kingery[25] for a number of other materials. For this purpose the tensile strength was assumed to be 65 pct of the rupture modulus[25,42]. Although there are unfortunately few data within identical temperature ranges for both beryllides and other brittle materials, some comparisons can be made:

1) Numerical values of R and R′ for beryllides at elevated temperatures are fairly high compared to other brittle materials. Thermal shock resistance of $ZrBe_{13}$ at 2300°F compares, for example, to that of MgO at 212°F. At low rates of heat transfer and at high temperatures, the thermal stress resistance of the beryllides is probably superior to that of most metal oxides and inferior to that of graphite, fused silica, and TiC cermet;
2) At high heat transfer rates the thermal shock resistance of the beryllides would be expected to compare to that of such materials as ThO_2, ZrO_2, and Al_2O_3; and
3) Based on values of R and R′, the beryllides should be better than they are. One possible explanation for the tendency of beryllides to crack upon cooling may lie in the presence of impurities in the beryllides.

Summary and conclusions

The intermetallic compounds formed between beryllium and the transition metal elements are hard, toxic, low density materials with good-to-excellent oxidation resistance and attractive nuclear properties. They are, without known exception, quite brittle even at temperatures approaching 2000°F. Several beryllides exist whose melting point lies above 3000°F, although none are known which melt above 3700°F. They retain their mechanical strength to rather high temperatures—Zr_2Be_{17} has a rupture modulus of 24,000 psi at 2750°F. Because the protective oxide which forms upon exposure to air is BeO the beryllides probably will have to be protected by some means against water vapor attack in air.

Despite their low temperature brittleness, there is some theoretical evidence that plastic slip deformation can occur in certain beryllides of the MBe_{12} type. Plastic deformation has been observed experimentally, but to a significant degree only above 2500°F.

$ZrBe_{13}$ has been extruded with some degree of success using heavy molybdenum cans and extrusion temperatures about 80 pct of the absolute melting point.

Thermal shock resistance has in practice and theory proved to be of the same order as that of some ceramic materials and inferior to graphite and fused quartz.

Those properties of beryllides which can be expected to be affected by the presence of impurities or by the fabrication technology have not really been evaluated thoroughly. This would include such properties as the mechanical strength, impact resistance, thermal conductivity, ductility, hardness and possibly oxidation resistance. It would seem proper to devote future effort to determining these intrinsic properties on high purity, controlled material.

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SINTERED HIGH-TEMPERATURE ALLOYS

This paper outlines some of the properties of alloys produced from atomized powders of nickel-chromium-cobalt and nickel-chromium-cobalt-molybdenum and describes certain aspects of further development work on sintered high-temperature alloys.

by V. A. Tracey, G. T. Poyner, and J. F. Watkinson

During the early development of high temperature alloys for gas turbines, the usual production technique was casting followed by forging to produce a wrought alloy. However, as development progressed, considerable attention was directed toward the development of alloys which possessed adequate high strength in the as-cast condition. This approach not only allowed the use of more highly alloyed compositions, but also reduced the cost of producing blades by employing precision casting techniques.

The first application of the powder metallurgical techniques in the high temperature field was originally suggested as a means of realizing in practice the theoretical advantages claimed for transpiration cooling. The application was abandoned due to insufficient strength of the porous material and to other difficulties associated with clogging of the pores. The development of sintered alloys continued, however, in the hope of using solid sintered alloys in shaped form and to extend useful alloy systems where forging was impossible and machining difficult. It was essential that any sintered alloys should possess properties at least equivalent to wrought or cast alloys used as turbine blade materials.

Early work at the General Electric Co. Laboratories, Wembley,[1,2] showed that by using appropriate mixtures of pre-alloyed powders produced by vacuum melting and ball milling, slightly lower stress-rupture properties could be obtained with sintered material than with cast vitallium of the same composition. Although this technique was a considerable advance, it was both expensive and laborious.

Since 1956, considerable emphasis has been placed on the development of suitable pre-alloyed, high-temperature alloy powders by the Birmingham Small Arms Co., Ltd. (B.S.A.) Group Research Center which resulted in the availability of alloy powders in a wide range of compositions. Pre-alloyed high-temperature alloy powders of the nickel-chromium-cobalt and nickel-chromium-cobalt-molybdenum types corresponding to the well-known high-temperature alloys *Nimonic 90* and *Nimonic 100*® were produced. These alloys are subsequently referred to as Types A and B respectively, and typical compositions of these alloys are presented in Table I. This paper outlines some of the properties of alloys produced from these powders, and describes certain aspects of the work on the further development of sintered high-temperature alloys.

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Sintering

Previous reports[3] have already described the techniques employed in the production of atomized pre-alloyed powders. These processes produce an irregularly-shaped powder with the following particle size distribution: 17 to 18 wgt pct from −100 to +200 mesh; 20 to 25 wgt pct from −200 to +300 mesh; and 48 to 61 wgt pct smaller than 300 mesh. The powders utilized in this investigation had similar pressing characteristics.

These alloy powders are best sintered in a vacuum of better than 1 μ and the temperature must be restricted to a narrow range if all porosity is to be eliminated. The sintering range for the Type A alloys was found to be between 2325° and 2380°F, whereas Type B alloys had to be sintered between 2355° and 2380°F. In each case, the upper temperature was the limit imposed by the formation of a liquid phase. Variations in the sintering time had a pronounced effect on the properties and the effects of varying the sintering period from 2 to 20 hr on the high-temperature properties were determined.

A study on the effect of sintering temperature on the density for a sintering time of 2 hr between 2280° and 2340°F for the Type B alloys and for 20 hr between 2190° and 2370°F for Type A alloys indicated that the sintered density of the two alloys usually falls within the range 8.0 to 8.1 g per cc for the Type A alloys and between 7.85 to 8.0 g per cc for the Type B alloys.

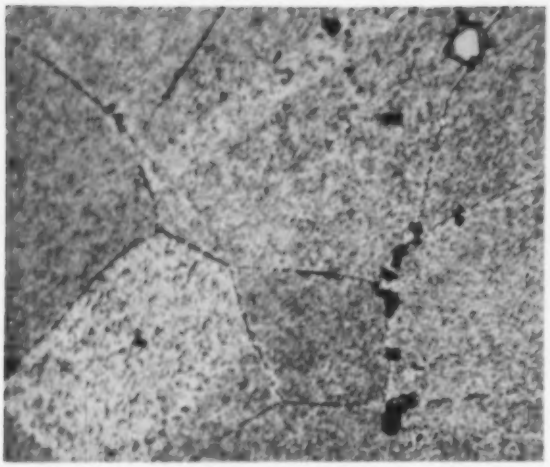

Fig. 1—Microstructure of sintered and heat treated Type A alloy. Etched electrolytically in glycerine-HF. 800X.

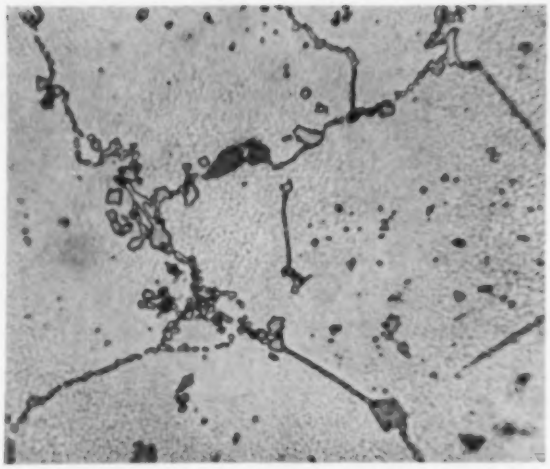

Fig. 2—Microstructure of sintered and heat treated Type B alloy. Etched electrolytically in oxalic acid solution. 1200X.

All specimens were machined from ½ in. square x 2 in. blocks which were sintered in pairs in special vacuum tube furnaces.[4]

Evaluation of physical properties

In all cases, the alloys received the following heat treatment: Type A alloys were heated for 8 hr at 1975°F, air cooled and then heated for 16 hr at 1290°F and air cooled. Type B alloys were heated for 1½ hr at 2190°F, air cooled and then heated for 16 hr at 1560°F and air cooled. The microstructures of the two alloys after their respective heat treatments are shown in Figs. 1 and 2.

Stress-Rupture properties: The stress-rupture results obtained on sintered material vary considerably with the density of the final product. The results obtained on Alloy A showed clearly that if the material contains some porosity, the stress-rupture properties are adversely affected. For this reason all properties apply to material sintered within the temperature range previously quoted—2325° to 2380°F. The time taken to obtain the maximum density, as judged by normal methods of determination, is of the order of 2 hr, but the stress-rupture properties improve with times in excess of this period independently of any measurable density changes. The increase in properties with sintering time can be divided into two sections; the first, where the properties increase rapidly followed by a second stage of gradual increase with only a slight improvement in stress-rupture properties. With −300 mesh powder, the change-over period occurs at about 4 hr, but with the coarser powder— −100 mesh—this transition is not so obvious. The higher stress-rupture lives, using −100 mesh powder are only obtained after sintering for times in excess of 12 hr.

The stress-rupture properties of the two alloys have been assessed after 20 hr at 2370°F, followed by the standard heat treatments. The stress-rupture properties at 1495°F and 1600°F for the sintered nickel-chromium-cobalt alloy is shown in Fig. 3, typical values for the wrought alloys are shown in the same diagram.

In all, fifteen Type B composition powders were investigated and it was found that generally the properties fell into two main groups represented by the curves in Figs 4 and 5.

The work of various American investigators, notably that of Freeman and his associates,[5] has shown that small amounts of boron and zirconium picked up from reactions between high-temperature alloy melts and the crucible material, significantly affected the stress-rupture properties. Insofar as these alloy powders were melted in magnesite linings prior to atomization of the melt, it seemed possible that variation in these minor constituents might also be responsible for the difference in stress-rupture properties between these two groups.

The addition of boron as chromium boride, to powders of the first group produced a considerable increase in these properties. To evaluate the effect of boron additions, a stress-rupture test was made with the alloy in the as-atomized condition and compared to data for a powder containing 0.05 pct B as chromium boride. The average stress-rupture life at 12 tons per sq in. and 1600°F for the as-atomized condition was 50 hr compared to 223 hr for the alloy with the boron addition. The same increase was not evident when chromium boride was added to powders of the second group. Still further increases in the stress-rupture properties were obtained when boron and zirconium were added simultaneously as zirconium boride containing 0.05 pct B and 0.20 pct Zr. Comparison of the stress-rupture tests with the as-atomized alloy indicated that the zirconium boride addition increased the average stress-rupture life to 541 hr.

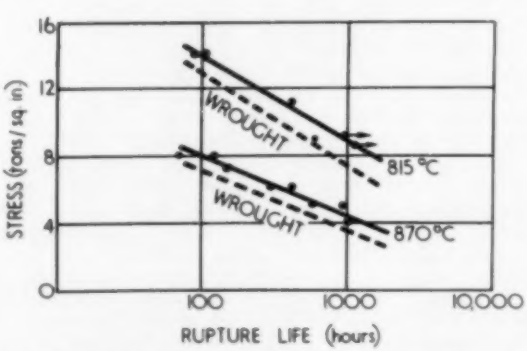

Fig. 3—Stress-rupture properties of the Type A alloys compared with wrought *Nimonic* 90.

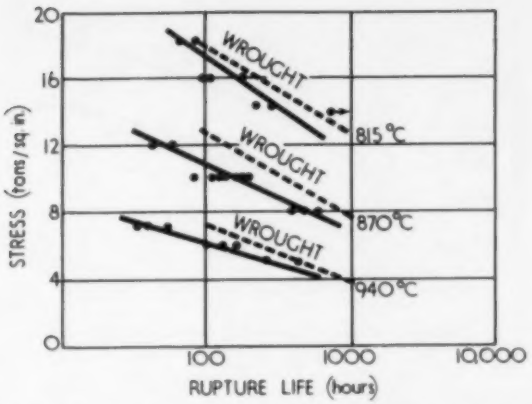

Fig. 4—Stress-rupture properties of the first group of Type B alloys compared with wrought *Nimonic 100*.

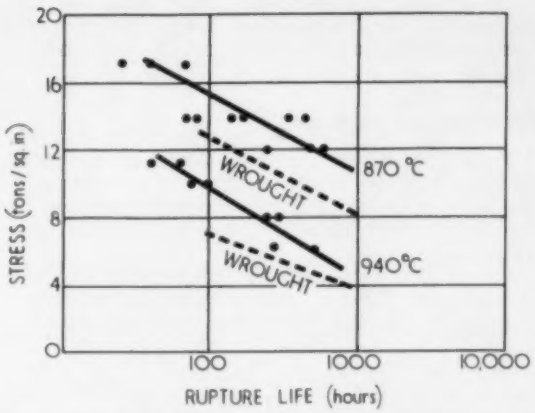

Fig. 5—Stress-rupture properties of the second group of Type B alloys compared with *Nimonic 100*.

These results confirmed the view that differences in the boron and zirconium contents were principally responsible for the varying behavior of powders in the two groups. Unfortunately, at that time, no facilities were available to determine the precise boron content of each powder and so it was not possible to obtain complete confirmation.

The influence of the zirconium and boron additions was confirmed using another alloy powder which did not contain any detectable amount of boron, by making additions of boron and zirconium as chromium boride, zirconium boride and/or zirconium carbide. The results showed that with this powder the maximum stress-rupture properties, when tested at 10 tons per sq in. at 1725°F occurred at 0.02 to 0.03 pct B and 0.2 to 0.3 pct Zr within the range of boron and zirconium contents investigated.

The elongation at rupture was considerably improved from the original value of 2.5 pct to over 10.0 pct over a wide composition range.

Fatigue properties: A limited number of alternating bending fatigue tests was carried out at 2500 cycles per min and 1600°F using machines described by Gresham & Hall.[6] The fatigue strengths are somewhat lower than those quoted for wrought *Nimonic 100*[7] and the fatigue properties appear to be independent of sintering time between 2 and 20 hr. Typical results for the Types A and B alloys, at 1600°F are given in Table II.

Table I. The Composition of the Nickel-Chromium-Cobalt Alloys and the Nickel-Chromium-Cobalt-Molybdenum Alloys

Element	Composition, wt pct	
	Alloy Type A	Alloy Type B
Carbon	0.05	0.25
Cobalt	19.00	20.00
Chromium	18.00	11.00
Aluminium	1.50	5.00
Titanium	2.50	1.50
Molybdenum	—	5.00
Nickel	Bal	Bal

Table II. The Fatigue Strength of the Type A and B Alloys at 1600°F

Stress Range, tons per sq in.	Cycles to failure, millions	Time, hr
Type A		
0 ± 14	5	33
0 ± 10	15	100
Type B		
0 ± 18	2	13
0 ± 16	6	40
0 ± 14	25	167

The fatigue properties of these alloys were generally about 20 pct below those of the equivalent wrought material, i.e. *Nimonic 90* and *Nimonic 100*. The effect of higher boron contents in the sintered materials was investigated by the addition of 0.04 pct B and 0.16 pct Zr as zirconium boride. The results obtained on one batch of powder showed a three-fold increase in the number of cycles to failure for an approximate increase of 10 to 15 pct on a stress basis.

Impact resistance: The impact resistance of the sintered materials of both alloys was lower than that of the wrought alloys. The room temperature impact value, as measured by the Izod notched testpiece, was of the order of 6.0-ft lb for the Type A alloys and 3 to 6-ft lb for the Type B alloys. The impact value of the latter alloy was improved to 10-ft lb by the addition of 0.04 pct B and 0.16 pct Zr.

Thermal shock properties: Some indication of the thermal shock properties was obtained from a limited number of tests on the Type B alloys using the N.G.T.E. method.[8] The material was tested after heat-treatment, at 2190°F for 1½ hr followed by 16 hr at 1560°F, and it was shown clearly that the thermal shock properties were considerably better than for a wrought material of a similar composition. Tests on material which had been over-heated during sintering showed that the thermal shock properties were considerably lower, probably due to the presence of a continuous grain-boundary phase. The improved thermal shock properties of correctly sintered material, were probably due to the fine and even grain-size of such material.

Grain-size characteristics: The grain-size of both types of alloys was shown to be dependent on the sintering time and the original particle-size of the powder.

The results obtained on a Type B alloy sintered between 2 and 20 hr at 2370°F is illustrated in Fig. 6 for −300 mesh powder, and also for a blend of 25 pct −100, +200 mesh; 25 pct −200, +300 mesh; and

50 pct of −300 mesh powders. The results clearly indicated that the grain-size of the sintered material increases with sintering time and powder particle-size.

Fabrication methods

Turbine blade sections were produced by hydrostatic pressing at pressures around 20 tons per sq in., and in one case cooling channels were produced using the General Electric Co. cadmium-wire technique.[6]

Slip-casting was investigated using a slip containing approx 25 pct water with 0.4 pct ammonium alginate as a binder. The presence of coarse powder made it difficult to obtain a suspension and, therefore, −300 mesh powder was used for all experiments. Both simple and complex shapes were produced, and under the conditions outlined, *green densities* obtained were of the order of 3.5 g per cc. The irregular particle shape of these powders was mainly responsible for this low *as-cast* density and spherically-shaped powders gave green densities of around 5.0 g per cc. The properties obtained from the sintered slip-castings were comparable with those obtained by normal pressing methods. The sintering of complex shapes, was always difficult and pre-sintering was necessary in order to impact sufficient strength and prevent cracking in the final stages of densification.

Although one aspect of the use of sintered material involves the production of a finished or near-finished component requiring the minimum of machining or grinding and polishing, other potential applications would involve some degree of forging.

Comparison tests at 1920°F and 2083°F have shown that the materials behave in a similar manner to the equivalent wrought alloys. In tests at 2083°F up to 50 pct deformation on slow compression was given to test-samples without producing cracking.

Type A alloys were forged at 2083°F with a 75 pct reduction in cross-section to produce ⅝ in. square bars, and both alloys were forged into turbine blades from sintered compacts at 2083°F.

The grain-size of sintered material in both alloys was found to decrease after all working operations, although no critical survey over small deformations, followed by heat-treatment, has yet been investigated.

The grain-size of forged bar, reduced 75 pct in cross-sectional area, decreased from 0.056 to 0.035 mm. There was also an indication that the stress-rupture properties were slightly reduced by forging.

Discussion

The first stage of the investigation was to show whether sintered high-temperature alloys could be produced from pre-alloyed powders, having high-temperature properties comparable with the conventional wrought and cast materials. The results presented indicate that in certain respects, the sintered materials offer distinct advantages, i.e. creep strength, fine grain-size, thermal shock and homogeneity, but the fatigue strength, ductility and impact strength are lower than for the wrought alloys. However, controlled additions of boron and zirconium improved these properties to, and above, a minimum acceptable level.

The level of properties in these sintered alloys enters the range only obtainable in wrought material by using vacuum-melting techniques. It appeared probable that further improvements could be obtained by some investigation into the compositions of the alloy powders, instead of using those compositions corresponding to known wrought alloys.

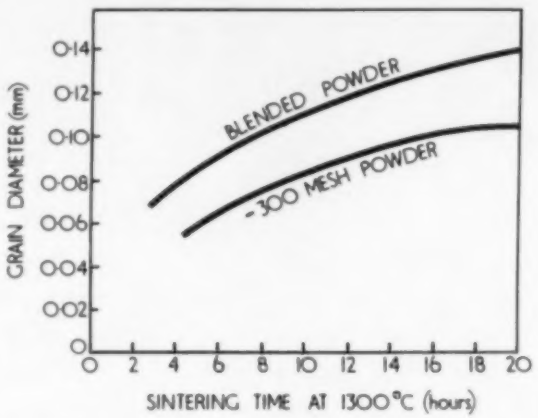

Fig. 6—Effect of sintering time and particle size on the grain size of Type B alloys.

Alloy improvement

There were indications from the previous work that the stress-rupture properties of Type B alloys could be improved by raising the total titanium and aluminum contents and that the introduction of tungsten with molybdenum as an additional solid-solution hardening-element would be beneficial.

Increases in the aluminum and titanium contents are not always desirable in wrought alloys because forgeability is markedly reduced. It was, however, appreciated that a searching appraisal of the effects of varying the compositions of the pre-alloyed powders was required, if the potentialities of the powder metal approach were to be exploited.

The number of experimental alloys which were required to cover the desired range was economically prohibitive because the minimum size melt which could be produced was 100 lb. It was decided that a number of master alloy powders should be produced which could be blended to give the required variations in the composition.

The compositions of the master alloy powders used are given in Table III.

Preliminary experiments centered on the effect of varying the molybdenum and the tungsten contents, together with differing proportions of titanium and aluminium on the stress-rupture properties of a 16 pct Cr, 20 pct Co, 0.25 pct carbon-nickel base alloy. The chromium level was fixed at 16 pct in these experiments because of the inferior oxidation resistance of materials containing 10 to 12 pct Cr.

Table III. Compositions of Masteralloy Powders

	Wt Pct							
Alloy	C	Cr	Co	Mo	W	Ti	Al	B
400A	0.28	15.5	21.6	9.86	—	1.8	4.6	—
401A	0.04	16.4	22.6	10.6	—	2.25	5.62	—
402A	0.31	15.5	21.6	—	9.35	1.55	4.9	—
403A	0.05	15.6	19.8	—	9.48	1.55	5.08	—
404A	0.30	16.7	20.1	—	—	1.95	5.54	—
405A	0.05	15.6	21.2	—	—	2.11	5.56	—
406A	0.09	15.6	19.6	—	—	1.95	5.58	0.3
407A	0.05	15.6	19.5	—	—	0.77	10.4	—
408A	0.01	15.3	22.8	—	—	10.0	—	—

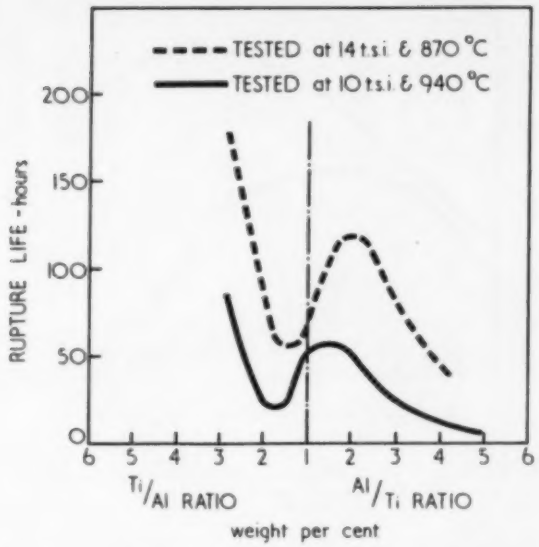

Fig. 7—Effect of the titanium-to-aluminum ratio on stress-rupture properties.

The zirconium and boron contents were held at 0.16 and 0.04 pct respectively in the initial work with a view to investigating their effect more fully in those alloy compositions which showed the greatest promise.

Effects of alloying

The effects of molybdenum and tungsten on the stress-rupture life were investigated at 1600°F and at 14 tons per sq in. (31,400 psi), using the following alloy base: 0.25 pct C, 2.0 pct Ti, 5.0 pct Al, 0.16 pct Zr, 0.04 pct B, 16 pct Cr, and 20 pct Co.

Under these conditions, additions of up to 7½ pct W produced a continuous improvement of the physical property while approx 4 pct Mo yielded the maximum improvement. In both cases the maximum life achieved was approximately 200 hr. The value for the 5 pct Mo alloy made in this way agrees well with the corresponding life for completely pre-alloyed Type B alloy, which suggests that the experimental approach, using mixed powders which do not differ greatly in composition, is a valid one.

A limited number of tests were carried out using combined additions of molybdenum and tungsten. Under the same test conditions, a 2½ pct Mo, 5 pct W alloy gave an average life of 700 hr and the 5 pct Mo, 2 pct W alloy 300 hr.

Other tests carried out at 1725°F and at 10 tons per sq in. (22,400 psi) suggested that the higher tungsten alloys containing between 4 to 7 pct W combined with a molybdenum content of the order of 2 to 4 pct showed the most promise.

The results of varying the titanium to aluminium ratio on the stress-rupture properties at 1600°F and 1725°F are summarized in Fig. 7 for the following alloy base: 0.3 pct C, 16.0 pct Cr, 20.0 pct Co, 5.0 pct Mo, 0.04 pct B, and 0.16 pct Zr. The results refer to alloys with a total aluminium and titanium content of between 7.5 pct and 9.0 pct and it is clear that either an aluminum:titanium ratio of around 2.0 or a titanium: aluminium ratio of 3.0 produces the strongest alloys, under these conditions.

The following composition was chosen as the basis for more detailed investigation: 0.3 pct C, 16.0 pct Cr, 20.0 pct Co, 2.5 pct Ti, 5.5 pct Al, 2.5 pct Mo, 5.5 pct W, and balance Ni; this alloy is now known as *Cosint 1000*®.

Several atomized powders corresponding to this composition were prepared. In addition, both boron and zirconium were added to certain of the melts. The results of the investigation of the properties of the sintered material can be summarized as follows.

Stress-rupture properties: The stress-rupture properties of the alloy were investigated under a load of 7 tons per sq in. (15,680 psi) at 1800°F. The properties of the alloy were markedly dependent on the boron and zirconium contents. Thus, a powder which contained approx 0.2 pct Zr and 0.10 pct B as atomized, gave a life of 120 hr while a powder which contained only 0.010 pct B as atomized had a life of 2 hr which increased to 46 hr with 0.2 pct Zr addition. Although the optimum stress-rupture properties are achieved at a boron content of 0.1 pct, the effect of this addition on other properties is significant.

A study on the effect of variations in the tungsten content of *Cosint 1000* suggested that the tungsten content should be closely controlled in the range 5 to 6 pct. From an investigation of the combined effects of aluminium and titanium it would appear that 5.5 pct Al and 2.5 pct Ti are the preferred values, and that in this alloy titanium and aluminium contents of over 8 pct are detrimental to the stress-rupture properties.

Room Temperature Impact Strength: The impact values of the Type B alloys described earlier were only 3 to 6 ft-lb. However, the impact properties of sintered *Cosint 1000* were much better, provided the boron content was limited to 0.07 pct. Optimum impact properties were attained with boron additions between 0.03 to 0.07 pct.

Tensile Properties: The addition of boron progressively reduces the ultimate tensile strength at room temperature but the tensile strength is increasingly better in the higher boron material as the test temperature is progressively raised above 1472°F.

Fatigue Strength: The fatigue properties of *Cosint 1000* have been determined at 1600°F and it was found that the fatigue properties appear unaffected with changes in the boron content from 0.03 to 0.11 pct B.

The composition of the alloy which gave the most suitable combination of properties was: 0.3 pct C, 16.0 pct Cr, 20.0 pct Co, 2.5 pct Mo, 5.5 pct W, 2.5 pct Ti, 5.5 pct Al, 0.2 pct Zr, 0.06 pct B, and balance Ni.

General discussion

It is clear that sintered high-temperature alloys can be produced from atomized pre-alloyed powders which compare favorably with the commercial alloys in present use. One special feature of the sintered alloys is the uniform fine grain-size which is relatively unaffected by heat-treatment and/or mechanical working. This is interesting, especially in view of the fact that the sintered alloys have

better creep-strength, but lower fatigue resistance, compared with the coarser-grained wrought alloys.

In this connection X-ray analysis of the non-metallic extracts from −300 mesh Type B and *Cosint 1000* powders showed no alumina, but alumina is known to be present in extracts prepared from alloys sintered from these powders. Coarser powders contain some alumina prior to sintering but in this case the percentage of alumina increases on sintering. The alumina is present as α-alumina and it was estimated that approx 0.3 pct was present in the sintered alloys.

It has not been possible so far to determine the location of the alumina particles in the sintered alloy. It is probable, however, that the alumina forms as a result of a reaction between the aluminium in the alloy-powder and either air, entrapped in the compact during pressing, or with adsorbed layers present on the irregular powder particles. It is generally observed that as the compacts are raised to the final sintering temperature the vacuum gauges show two outgassing peaks, one at 570°F and the other at 2100°F to 2190°F. Specimens prepared from −300 mesh powder and sintered at 1920°F do not contain alumina, while those sintered at 2190°F and above, do contain alumina. It seems likely that the second outgassing represents the removal of adsorbed gas, and that alumina forms at this time.

In our view, the stability of the grain-size is associated with the presence of alumina: and, more important, it is possible that a dispersion of alumina, though not present to the extent normally required for dispersion hardening, does confer some of the attributes of a dispersion-hardened alloy on these sintered materials. This could explain the higher stress-rupture and lower fatigue properties of sintered alloys compared with the same alloy in wrought form.

Boron and zirconium produce a great improvement in the properties of sintered alloys. Similar improvements are produced by small additions of these elements to wrought alloys, though two significant differences are apparent:

1) The amounts of boron and zirconium, which produce optimum properties in wrought alloys are much less than those required in sintered materials; and
2) The extent of the improvement in the wrought alloys, while marked, is not as great as in the sintered materials.

The work of Freeman and his associates[20] presents a clear and convincing picture of the manner in which boron and zirconium improve the properties of wrought and cast high-temperature alloys. Their findings can be summarized as follows:

1) Boron and zirconium inhibited and delayed the agglomeration of $M_{23}C_6$ type carbide in grain-boundaries normal to the applied creep-stress. The agglomeration of carbide is accompanied by depletion of the γ′ [$Ni_3(Al{\cdot}Ti)$] from the adjacent matrix and microcracks formed at the interface between the $M_{23}C_6$ carbide and the depleted matrix initiate premature failure;

2) The number of γ′ depleted zones and microcracks is lower in those alloys which contain zirconium and boron under comparable conditions of equal strain and time, after creep-testing at 1600°F. The secondary creep rates decreased with additions of boron and zirconium and both elements prolonged the third stage of creep. The appearance of microcracks was delayed from late in the primary stage of creep for boron and zirconium-free materials, until well into the tertiary stage for alloys containing the optimum amounts; and

3) Boron was more effective than zirconium, because in addition to inhibiting the formation and agglomeration of carbides at the grain boundaries it favored carbide precipitation within the grains as M_6C rather than $M_{23}C_6$.

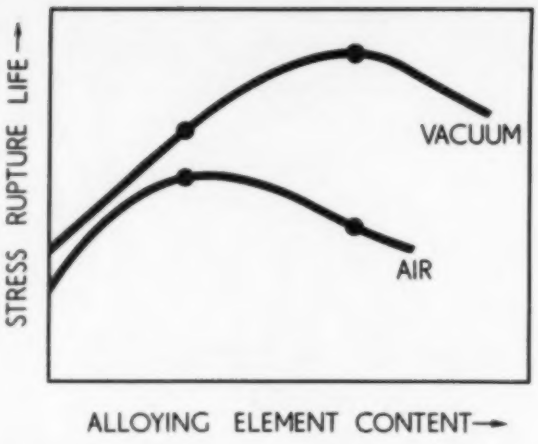

Fig. 8—Variation of stress-rupture life with alloying content for vacuum-melted and air-melted alloys.

It seems probable that equilibrium segregation of the boron and zirconium atoms to the grain boundaries reduces the tendency for carbide agglomeration (leading to microcrack formation) and effectively encourages the precipitation of carbide within the grains.

It follows that the sintered alloys which are much finer grained probably contain a higher density of vacancies and misfit regions than wrought alloys, would require higher additions of boron and zirconium to occupy and *neutralize* the favored sites for the nucleation and agglomeration of $M_{23}C_6$ carbides at the grain boundary.

On the basis that the role of boron and zirconium is essentially the same as in wrought alloys, but due to the greater grain-boundary area (and possibly a higher concentration of vacancies at the grain-boundary), higher percentages of these elements are required to inhibit those structural changes which lead on to failure. It is possible moreover, that boron (and zirconium) may influence the manner in which alumina forms, by influencing its formation at grain-boundaries. These conjectures underline the need for a more fundamental examination of these sintered materials, as distinct from *ad hoc* development, and especially for electron micrograph studies of the alloys.

While combined additions of tungsten and molybdenum produce some improvement in strength compared with Type B alloys, the extent of that

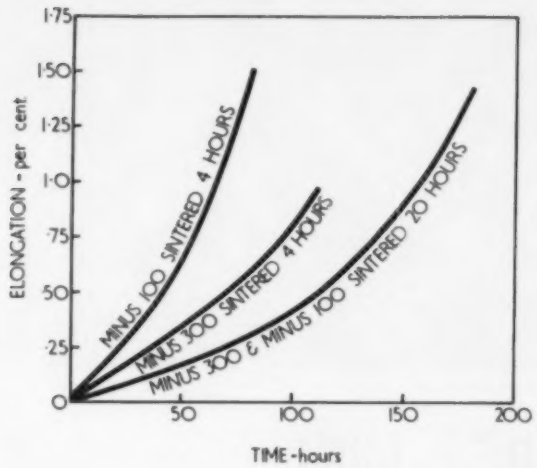

Fig. 9—Effect of sintering time and particle size on creep rate.

improvement is only minor compared to the combined effects of boron and zirconium. Further work has shown that a number of combinations of tungsten and molybdenum yield similar properties to *Cosint 1000* provided that the contents of the other alloying elements are adjusted for each alloy. It was found in the case of Type B alloys containing 5 pct Mo (and no tungsten) that considerable improvements could be obtained by raising the aluminium content to 7.0 pct. Unfortunately, corresponding improvements are not produced in the *Cosint 1000* alloys. In general, increased contents of the alloying elements, e.g. Mo, W, Ti, Al, Cr, and Co above the levels found in air-melted alloys do not produce any further improvements in the stress-rupture properties of the sintered alloys. Attention is therefore being currently directed to the production of vacuum-melted atomized powders. Some support for this approach can be found in a recent paper[11]. Gittus points out that in air-melted alloys there is an optimum alloy content above which the properties decline. The principal virtue of vacuum-melting lies not in the improvements produced in those compositions which possess the optimum properties as air-melted, but rather that the peak for vacuum-melted stock is only reached with a considerably greater alloy content, and that the peak level of properties is much greater as illustrated in Fig. 8. The maximum level of properties achieved for air-melted wrought alloys was approx 60 hr at 1725°F and at 7 tons per sq in. (15,680 psi) whereas the current optimum for vacuum melted wrought alloys is claimed as 30 hr as 7 tons per sq in. (15,680 psi) at 1900°F.

The sintered alloys, prepared from air-melted alloys, give a maximum of 100 to 200 hr under 7 tons per sq in. at 1800°F, which is considerably better than their air-melted wrought counterpart. There appears however, but slight prospect of increasing this level of strength significantly and the main interest lies in determining whether the application of vacuum techniques will produce corresponding improvements in the sintered alloys. One further point on alloy development is that it is generally accepted that the outstanding high-temperature properties of nickel-base alloys hardened with titanium and aluminium lies in the close relationship and matching between the lattice parameter of the precipitate and that of the matrix of the alloy. It should be possible using blended heats of pre-alloyed powders to achieve a close composition control which would enable exact matching (and it is presumed maximum properties) to be maintained.

The influence of sintering time on the stress-rupture properties is worthy of comment. The results showed that when using —300 mesh powder, although maximum density was reached after 2 hr, the stress-rupture properties reached a maximum value after 4 hr. In the case of —100 mesh powder, the same density was achieved after 2 hr, but at least 12 hr sintering was required to produce the same level of creep-strength; despite the fact that being coarser grained, this material should be stronger than the alloy made from —300 mesh powder.

It is suggested, as a basis for discussion, that this is due to variations in the rate of removal of vacancies from the different grain-sized materials. Atomized powders, produced by the disintegration and immediate quenching of a liquid stream of metal by high-pressure water jets, should possess a more disordered structure. The longer sintering time required to achieve the same life in the coarser grained alloy may be due to a higher rate of defect removal and *healing* of the disordered structure by the increased grain-boundary areas—which normally act as sinks—in the finer grained alloys. Some suport for this view is shown by the creep curves in Fig. 9 which refer to —100 mesh and —300 mesh powders sintered for 4 and 20 hr. The rupture life is much less for the —100 mesh powder sintered for 4 hr and this material has a much higher secondary creep-rate than the other alloys.

Conclusions

The use of prealloyed powders for the production of sintered high-temperature alloys offers certain advantages, and may lead to the development of materials with better properties than the vacuum-melted and cast alloys curently available.

The eventual use of these materials, as for instance in gas-turbine blading, will depend not only on economic considerations. They offer some advantage in the manufacture of small blades currently machined from bar stock.

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COPPER SEGREGATION AT THE LAKE SHORE MINE

A segregation process to concentrate ores not amenable to conventional floatation concentration or sulfuric acid leach treatment is described, and results are given for pilot-plant results over a six-month period.

by G. A. Freeman, Carl Rampacek, and L. G. Evans

A large aggregate tonnage of oxidized and mixed oxide-sulfide copper ore of the southwestern US is not amenable to conventional flotation concentration or sulfuric acid leach treatment. Most of the ores contain chrysocolla as the principal oxide copper mineral, for which no successful commercial flotation method has yet been developed. Acid leaching some ores is not feasible since they contain substantial quantities of calcite and other acid-consuming constituents. Other ores decrepitate during leaching or contain excessive slime and clay minerals which cause plugging of the ore beds.

Research at the Federal Bureau of Mines' Tucson Metallurgy Research Laboratory over the past several years demonstrated that the segregation process had merit for treating oxidized and mixed oxide-sulfide copper ores regardless of the gangue constituents present, or the physical characteristics of the feed.[1,2]

Small-scale pilot-plant tests performed by the Bureau of Mines and by Transarizona Resources, Inc., further demonstrated that the process was applicable to the highly-calcareous and iron-bearing oxide ore occurring in the Lake Shore deposit near Komelik, Ariz. Based on test results, the company began erection of a 175 tpd experimental segregation plant to further test the process in December 1959. Construction of the plant was completed in May 1960, and shakedown operations were started in June 1960. This paper describes the Lake Shore operations and summarizes the segregation plant results obtained over a six-month period ending in November 1960.

Briefly described, the segregation process comprises heating an oxidized or mixed oxide-sulfide copper ore with a halide salt and a carbonaceous material, such as coke or coal, at approx 1400° to 1500°F. Segregation produces fine metallic copper which can then be recovered by conventional copper sulfide flotation methods. Although numerous reactions are involved in the process, basically the copper is volatilized from the copper minerals as cuprous chloride and then reduced to metallic copper on the surfaces of the carbonaceous particles. The fine flakes often agglomerate forming nodules as long as ¾-in. and ½-in. in diam. These nodules, however, immediately fall apart with agitation. A more complete discussion of the process and its application to oxide and mixed oxide-sulfide copper ores is given in earlier reports.[1,2]

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Lake Shore mine

Lake Shore mine is located at an elevation of about 1800 ft on the southwest piedmont of the Slate Mountains about 30 mi south of Casa Grande and 3 mi east of Komelik, Ariz. A shaft was sunk in the deposit in the period 1880 to 1884. Ore was produced sporadically from several shafts and three development levels in the deposit in the period between 1884 and 1929, when the last reported production was made. Total production from the mine is reported to have been about 280,000 lb of copper.[3]

Although previous *high-grading* operations employed underground mining, the deposit is now being mined by the open-pit method. Part of the north ore body has been stripped of alluvium down to a depth of about 30 ft to expose enough ore for several years of operation. Drill holes are spaced in a pattern of 5 to 6-ft centers; use of ammonium nitrate readily breaks the ore to about 10-in. in size. Loading is done by a 1-yd, front-end loader. The ore is hauled in 10-ton trucks to the segregation plant about 1500 ft southwest of the pit.

The ore being treated is a reddish-colored, magnetite-bearing, quartzite which has been fractured and recemented with quartz, chrysocolla, hematite, calcite, and chlorite material. The ore is devoid of sulfides, and at times, massive magnetite, enriched

in chrysocolla, and considerable calcite is encountered. The grade of minable ore varies from about 1.5 to 2.25 pct Cu. Detailed development work by rotary drilling has assured the mining and delivery of a consistently uniform feed to the plant.

Segregation-Flotation plant

The segregation-flotation plant has a daily capacity of about 175 tons of ore containing 3 to 4 pct moisture, the capacity being limited by the segregation furnace. A flowsheet of the plant as of November 1960, is shown in Fig. 1.

Power is supplied at 24,900 v from the lines of the Trico Electric Co. about 2 mi southwest of the deposit. A 750-kva transformer substation at the plant site drops the voltage to 440 v for plant operation, and a standby diesel-electric plant provides emergency power for the segregation furnace and calcine cooler during power failures.

Water from a 251-ft well about 2.5 mi west of the plant is delivered to the plant through a 5-in. pipe line by means of 30-hp pump. Pumping facilities are available to produce enough water to treat as much as 500 tons of ore per day. In addition to the primary water source, provisions are being made to recover water from the tailing disposal pond and thickeners. It is anticipated that 50 to 60 pct of the water will ultimately be recovered.

Natural gas, purchased from the Southwest Gas Co. is received at the property through a 4-in. line at 150 psig. The high pressure gas is reduced to 20 lb for plant use.

The ore is dry-crushed to −6 mesh for segregation. Coarse crushing is accomplished in a 16x24-in. jaw crusher set at 2 in. operating in open circuit. The jaw crusher discharge is conveyed and crushed in a 5-ft Tornado impact crusher in closed circuit with a 6 mesh vibrating screen. Rapid and efficient reduction is obtained in the crusher so that the circulating load is light. The undersize particles from the 6 mesh screen are conveyed to a 400-ton, fine-ore storage bin.

Crushing is carried out on the day shift. About 60 tons of ore per hour can be crushed to −6 mesh.

A typical screen analysis of the −6 mesh segregation furnace feed is given in Table I.

Segregation Furnace

The segregation reactor measures 54-in. inner diam x 48 ft long with the ends coned to 30 in. inner diam. The segregation unit is fabricated of ½-in. rolled and welded, type-316 stainless steel plate. A refractory brick-lined, natural gas-fired furnace, 31 ft long, encloses part of the reactor. About 8 ft of the shell extends from the furnace at the feed end and 8 ft extends at the discharge end. Riding rings attached to the reactor shell on either side of the furnace support the reactor. The discharge end outside of the furnace is completely insulated with 4½-in. thick magnesite block to reduce radiation losses to a minimum. Staggered, 3/16-in. stainless steel lifters 3 in. high and 24 in. long are attached to the inside of the reactor throughout the 54-in. diam section to facilitate mixing of the ore charge as it passes through the unit. The reactor is driven at 4 rpm by a 20-hp motor connected to a speed reducer which drives a ring gear attached to the reactor shell at the feed end. The furnace has a 74-in. inner diam and is equipped with 14 natural-gas burners complete with spark ignited pilots and auxiliary air and gas valves. The gas burners are located at the side and bottom of the furnace, firing tangentially to the rotating shell. Two dampered flues attached to the top of the furnace and near each end conduct the exhaust gases to a single 24-in. x 40-ft stack which discharges into the atmosphere.

The furnace is equipped with a complete temperature control system incorporating flame protection features and safety shut-off valves which automatically cut off the natural gas should the external shell temperature exceed a predetermined figure.

The outside temperature of the reactor tube is measured by five thermocouples equally spaced and riding on top of the reactor shell in the heated zone. Another thermocouple indicates the stack gas temperature. These temperatures are recorded on a 8-point strip chart recorder. A sixth thermocouple, located in the ore bed about 21 ft from the discharge end of the reactor is the principle fur-

Table I. Screen Analysis of Segregation Feed

Screen Size, Mesh		Wt Pct
	+6	—
−6	+8	4.6
−8	+10	7.9
−10	+20	18.7
−20	+35	16.9
−35	+48	7.3
−48	+65	7.3
−65	+100	7.5
−100	+150	7.1
−150	+200	4.7
−200	+270	4.6
−270		13.4
Composite		100.0

Table II. Screen Analysis of Tailing

Screen Size, Mesh		Wt Pct
	+48	0.4
−48	+65	1.0
−65	+100	5.3
−100	+200	25.0
−200		68.3
Composite		100.0

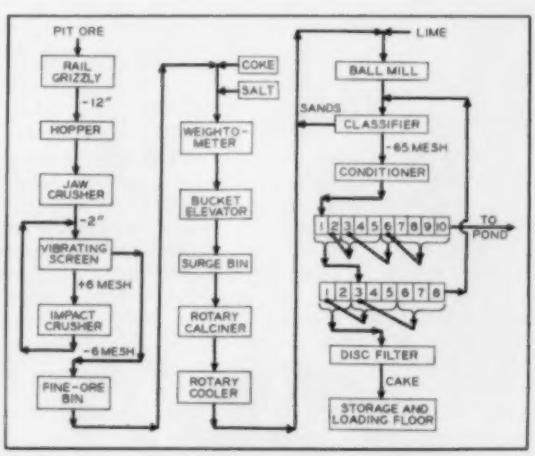

Fig. 1—Flow sheet of the Lake Shore copper segregation plant as of November 1960.

nace control. This thermocouple is connected to a circular chart potentiometer which operates a pneumatic control valve which increases or decreases the fuel to the burners, depending on the temperature of the charge. A second thermocouple is installed in the ore bed about 15 ft from the discharge end and also is recorded on the 8-point recorder.

The −6 mesh ore from the fine-ore bin and the required quantities of −20 mesh commercial salt and −48 mesh petroleum coke are fed on a 18-in. x 20-ft long conveyor belt using Syntron feeders. Some mixing of the salt, coke and ore is achieved on the conveyor belt by means of chains and rubber fingers suspended above the belt which drag in the ore ribbon as it passes beneath. The partly mixed feed discharges into a bucket elevator and then into a 10-ton surge bin ahead of the segregation furnace. Mixing of the ore, salt and coke is completed in the elevator. It was found that a thorough mixing of the salt and coke with the ore was very important.

The salt-coke-ore mixture is fed to the segregation furnace at a rate of about 7.5 tons per hour by means of a variable speed 9-in. spiral screw feeder connected to the bottom of the surge bin and extending about 3 ft into the reactor. A ring seal between the screw feeder and the reactor prevents escape of the reaction gases. An ore column is maintained in the furnace surge bin for air seal.

The temperature of the reactor charge is increased as rapidly as practicable; the outside shell temperature, or more likely the flame temperatures at shell surface are 1500° to 1700°F. The desired reaction temperature of 1400°F is reached in the ore bed at a point about 22 ft from the feed end and is maintained at this temperature until the ore leaves the heated section. The temperature then drops to about 1300°F as it moves into the insulated section toward the discharge end.

The normal bed in the reactor occupies a cross sectional volume of about 20 pct. Under the operating conditions employed, the contact time of the ore charge at the reaction temperature of 1400° to 1500°F is about 18 min.

The segregated calcine discharges from the reactor at approx 1100°F through a vented hood attached to the shell and feeds into a 4x44-ft Baker-type cooler. Moderately gas–tight connections are maintained between the reactor hood and cooler by means of ring seals. Absolutely air–tight connections are not required because the segregation reaction produces steam and reaction gases which develop a positive pressure and prevent entry of air into the unit.

Grinding and flotation

The cooled calcine discharges from the cooler at 200°F onto a 18-in. x 68-ft conveyor belt which feeds into a 6x12-ft Allis-Chalmers ball mill operating in closed circuit with a Dorr Duplex Classifier. The grind is controlled to give a −65 mesh classifier overflow. The classifier overflow, containing about 25 pct solids, is conditioned in a 5x5-ft slow-speed conditioner using potassium amyl xanthate, methylisobutyl carbinol and enough lime to establish a pulp pH of 11.5. The conditioned pulp is floated in 10 No. 24 Denver cells. A rougher concentrate is recovered from the first two cells and gravity-fed to the cleaning section. Froth from the next three rougher cells is returned to No. 2 rougher. Froth from No. 6 cell is returned to No. 3 rougher and a scavenger froth obtained from the final four roughing cells is combined with feed to the No. 6 rougher cell. The final tailing is pumped to a tailings pond 1000 ft away. The average screen analysis of the tailings is given in Table II.

The cleaning section consists of eight No. 18 special Denver cells. The rougher froth is cleaned twice using three cells in the first stage and two in the second. The final flotation concentrate is filtered on a 6-ft disc filter and stored in a bin for periodic shipment to the smelter. The first cleaner tailing is refloated in three additional cells to produce a scavenger concentrate which is combined with the froth from the roughing circuit. These products return to the head of the cleaning circuit, and the tailings from the middling refloat are returned to the Dorr classifier.

Results

Plant operations have been as good as, and in some instances have exceeded, the results obtained in the laboratory and pilot-plant research. A summary of results obtained during a 27 day period during the month of November 1960, are given in Table III. The grade of concentrates produced has consistently improved since the start of operations, and in some cases has approached 65 pct Cu. Copper recoveries also have improved, and the gas and reagent requirements have gradually been reduced. The gas consumption will be reduced considerably with the installation of pre-heaters using the waste stack gases.

Considerable experimentation was conducted in the plant and at the Federal Bureau of Mines' station in Tucson during the six months of operation. A number of changes in the original furnace design and simplification of the hot and cool calcine handling system were made before settling on the final flow sheet.

Based on the plant operation and results, the Lake Shore operations now are being expanded to about 750 tpd. The company will install two new furnaces, each of which will have a capacity of 10 tons of ore per hour. The capacity of the experimental reactor also is being increased to 10 tons by making a number of modifications in both the reactor and furnace design. Three pre-heaters will also be incorporated into the flowsheet to take advantage of waste heat.

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Table III. Summarized Results of Plant Operations, November 1 to November 27, 1960

Product	Assay, Pct Cu	Fe	CaO	SiO_2	Al_2O_3	Distribution, Pct Cu
Heads	1.81	13.7	—	—	—	100.00
Concentrate	50.09	3.2	2.6	13.3	0.8	87.15
Tailing	0.23	—	—	—	—	12.85

Reagents		
NaCl	lb/ton ore	28.0
Coke	lb/ton ore	18.0
CaO	lb/ton ore	4.8
Potassium amyl xanthate	lb/ton ore	0.5
Methylisobutyl carbinol	lb/ton ore	0.1
Natural gas	Btu/ton ore	1,900,000

HISTORY OF THE COKING INDUSTRY IN THE UNITED STATES

Part II of a four-part article

In last month's JOURNAL OF METALS, *Part I of this article described the early coke processes employed in the US. This month the article presents a history of the beehive oven era.*

by C. S. Finney and John Mitchell

The introduction of ovens for the production of metallurgical coke is believed to be due to L. L. Norton who operated an iron foundry in the vicinity of Connellsville, Pa. Persuaded by his foreman, an English immigrant named Nickols from Durham, L. L. Norton put up a 12-ft square oven which produced coke in 1833. The coal used was taken from a local mine at Mounts Creek. The oven seems to have been used in conjunction with the customary method of coking in mounds. It was in the Connellsville district also, in 1841, that two carpenters, Provence McCormick and James Campbell, formed a partnership with John Taylor, a stone mason, for the manufacture and sale of oven coke. The task of the mason was to construct the ovens, while the carpenters were to build the arks by which the coke could be taken by water to the market at Cincinnati. The following account of the enterprise was given by McCormick: "James Campbell and myself heard in some way that I do not now recollect that the manufacturing of coke might be made a good business. Mr. John Taylor, a stone mason, who owned the farm on which the Fayette coke works now stand, and who was mining coal in a small way, was spoken to regarding our enterprise, and proposed a partnership—he to build the ovens and make the coke and Mr. Campbell and myself to build a boat and take the coke to Cincinnati, where we heard there was a good demand. This was in 1841. Mr. Taylor built two ovens. I think they were about 10 feet in diameter. My recollection is that the charge was 80 bushels. The ovens were built in the same style as those now used, but had no iron ring at the top to prevent the brick from falling in when filling the oven with coal, nor had we any iron frames at the mouth where the coke was drawn. The top and mouth had to be repaired when they fell in. In the spring of 1842 enough coke had been made to fill two boats 90 feet long—about 800 bushels each—and we took them to Cincinnati down the Youghiogheny, Monongahela, and Ohio, but when we got there we could not sell. Mr. Campbell, who went with the boats, lay at the landing some two or three weeks, retailing out one boatload and part of the other in small lots at about 8 cents a bushel. Miles Greenwood, a foundryman of that city, offered to take the balance if he would take a small patent flour mill at $125.00 in pay, which Mr. Campbell did. He had it shipped here. We tried it, but it was no good, and we sold it to a man in the mountains for $30.00, and thus ended our coke business."

So successful did the coke subsequently prove to be in use that the three partners were asked to deliver more. Evidently they had had enough of the coke business, however, for they refused to have anything more to do with it. Few ovens were built between 1841 and 1855, and it is reported that in the latter year, "there were only 26 coke ovens along the river above Pittsburgh". Successful coke makers of these years included Mordecai Cochran, Richard Brookius, and Colonel A. M. Hill. It was the use of coke in 1859 in the Clinton furnace erected by Graff, Bennett and Co. in a plant on West Carson Street, Pittsburgh, that brought the real beginning of the coke-iron era in America. Here the successful use of Connellsville coke as a blast-furnace fuel was demonstrated beyond all possible doubt, and from the year 1859 the coking industry expanded tremendously.

The era of beehive coke ovens

During the latter half of the nineteenth century and the early years of the twentieth, the major percentage of metallurgical coke produced in the United States came from beehive ovens. It was not until 1893 that the first battery of by-product ovens came

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Beehive coke ovens in 1893.

into operation (at Syracuse, N. Y.), and only in 1919 did the total production of by-product coke first exceed that from beehive ovens. Peak output of beehive coke occurred in 1916 when 35,464,224 tons were produced as opposed to the 19,069,361 tons carbonized in by-product ovens.

The Connellsville district of Pennsylvania was not only the birthplace of the oven coking industry in the US, it was in this region also that the most extensive growth of beehive coking operations took place. There were two reasons for the rapid development of the coke industry at Connellsville. First, enormous quantities of excellent coking coal were readily available from the Pittsburgh seam of the Connellsville basin. Secondly, adequate rail and water transportation facilities allowed the coke to be shipped as far west as California, south to New Orleans, and to such eastern cities as New York and Baltimore.

In the special report on the Coke Manufacture of the Youghiogheny River Valley issued in 1875 as part of the Second Geological Survey of Pennsylvania, the Pittsburgh Coal Bed in the Connellsville Basin was described as occupying a trough 3 mi wide and 50 mi long, and giving from 8 to 9 ft of workable coal that was soft, easily and cheaply mined, and yielded a coke of unusual excellence. At the time of the Survey, the coal could be mined, coked, and loaded on cars at the ovens for $0.0275 per bushel, or $1.37 per net ton.

A typical analysis of the Pittsburgh coal then mined at Connellsville was given as: Moisture—1.3 pct; Volatile Matter—30.1 pct; Fixed Carbon—59.6 pct; Ash—8.2 pct; and Sulfur—0.8 pct.

Owing to its columnar structure and friable nature, Connellsville coal was readily broken during mining and handling, and thus arrived at the coke plant in comparatively small, uniform pieces. Since it was also free from any considerable impurities, it could be charged to the beehive ovens without any form of preparation.

As already noted, the development of the Connellsville coking industry was materially assisted by its favorable location with respect to transportation. By means of the Youghiogheny, Monongahela, Ohio and Mississippi rivers, the celebrated coke of Connellsville could be marketed in cities such as Cincinnati, St. Louis, and even New Orleans. In addition to this great river network, however, the coke region was served by four railways; the Pittsburgh, Washington and Baltimore (Pittsburgh and Connellsville) main line ran along the north bank of the Youghiogheny, Pittsburgh being 57 mi and Baltimore 287 mi from the town of Connellsville; the South West Pennsylvania Railroad connected with the Pennsylvania Railroad at Greensburg, 24 mi north of Connellsville; and short branch lines 10 and 9 mi in length, respectively, linked Connellsville with the coke works south as far as Uniontown and north to Mount Pleasant. Thus it was that from this small area of undulating country in Westmoreland and Fayette counties in southwestern Pennsylvania, Connellsville coke went out to Ohio, Milwaukee, Chicago, Omaha, Salt Lake City, and California in the west; to the Gulf Coast in the south; and to New England, New York City, Philadelphia and Baltimore on the Atlantic coast.

By 1875 there were 3578 beehive ovens in the Connellsville Basin, capable of producing 26,000 tons or 1,302,600 bushels of forty-eight hour coke when operating full time. Details for the four districts comprising the Connellsville region are as follows:

District	No. of Works	No. of Ovens	Weekly Shipments in Cars of 600 Bushels
Fayette County Branch of Pittsburgh & Connellsville R.R.	7	646	375
Mount Pleasant Branch of Pittsburgh & Connellsville R.R.	21	1349	880
Pittsburgh Connellsville R.R.	8	953	562
South West Pennsylvania R.R.	9	630	374
Total	45	3578	2171

The beehive coking industry was not, of course, confined to the state of Pennsylvania, much less to

Table I. Analysis of Coals of Various Areas at the Close of the 19th Century

Appalachian Coals

State	Moisture, Pct	Volatile Matter, Pct	Fixed Carbon, Pct	Ash, Pct	Sulfur, Pct
Pennsylvania—East	1.73	23.89	67.03	6.69	0.66
Pennsylvania—West	1.70	39.15	46.66	10.52	1.97
Ohio	1.58	41.86	51.44	5.12	2.64
West Virginia—East	1.52	19.81	72.71	5.20	0.76
West Virginia—West	1.52	37.86	53.37	6.03	1.22
Kentucky	1.80	33.00	60.10	5.10	0.65
Tennessee	1.30	32.51	59.33	5.82	0.84
Alabama	1.65	32.48	60.15	4.62	0.90
Eastern[a] and Western[b] Interior Coals					
[a]Illinois	2.08	37.10	52.17	7.02	1.63
[a]Indiana	2.88	40.98	50.70	3.46	1.88
[b]Kansas	3.25	40.96	43.98	10.71	1.10
[b]Missouri	6.50	37.71	42.17	10.56	3.06
Rocky Mountain[c] and Pacific Coast[d] Coals					
[c]Colorado	0.82	37.25	55.72	6.00	
[c]Montana	1.02	38.01	48.20	11.87	Not
[c]New Mexico	6.66	40.13	45.56	7.65	Given
[d]Washington	3.26	35.36	57.58	3.80	0.10

Table II. United States Coke Production During 1896

	Coke Production, Net Tons
Appalachian Region	
Pennsylvania (includes New York)	7,356,502
West Virginia	1,649,755
Alabama	1,479,437
Tennessee	339,202
Virginia	268,081
Ohio	80,868
Georgia	53,673
Kentucky	27,107
Total	11,254,625
Eastern Interior Region	
Wisconsin	5,332
Indiana	4,353
Illinois	2,600
Total	12,285
Western Interior Region	
Indian Territory	21,021
Kansas	4,785
Missouri	2,500
Total	28,306
Rocky Mountain Region	
Colorado	343,313
Montana	60,078
New Mexico	24,228
Utah	20,447
Wyoming	19,542
Total	467,608
Pacific Coast Region	
Washington	25,949
Grand Total	11,788,773

the Connellsville district. By the close of the 19th Century, beehive ovens were at work in the following areas: the Appalachian region in the coal fields of Pennsylvania, Virginia, West Virginia, Ohio, Tennessee, Georgia, Alabama and eastern Kentucky; the Eastern Interior region in the fields of Illinois, Indiana and western Kentucky; the Western Interior region in Kansas, Missouri and Oklahoma; the Rocky Mountain province, including Colorado, Montana, Utah and New Mexico; and the Pacific coast province in the fields of Washington.

Typical analyses for the coals of these regions were given by John Fulton in his book *Coke* published in 1905 and are listed in Table I. Important as they may have been locally, the coke-producing areas other than the Appalachian region were of limited national significance. Either, as in the case of the Eastern and Western Interior regions, the coals were much less well suited to beehive operations than the strongly coking Appalachian coals; or alternatively, as in the case of the Pacific coast fields of Washington, coking coals were only found in small areas.

From Table II which illustrates the coke production in the US during the year 1896, it can be seen that of a total output of 11,788,773 net tons (including 83,038 tons of by-product coke) 11,254,625 tons were produced in the Appalachian region, Pennsylvania alone being responsible for more than 7 million tons. The total output for all the other coke-making regions combined was only 534,148 tons.

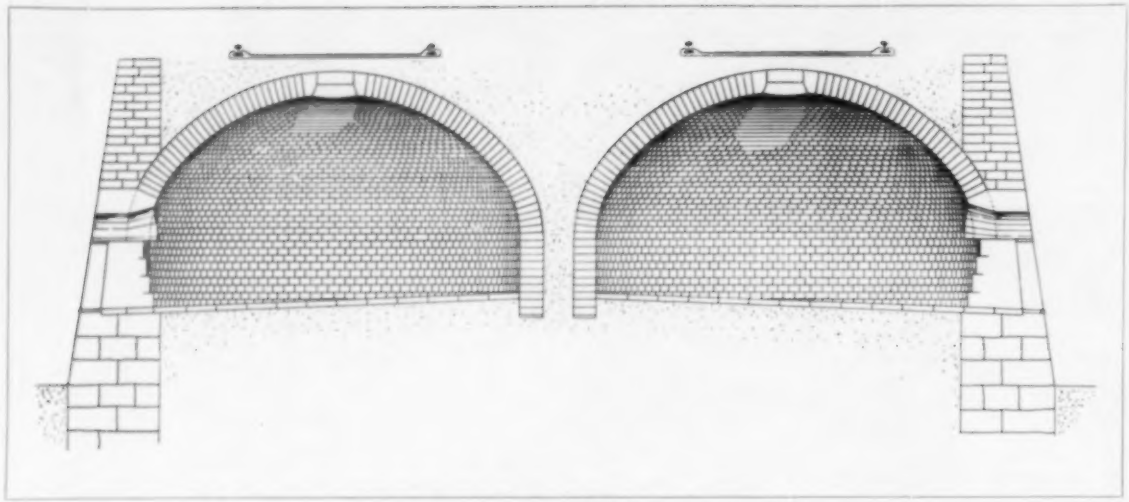
The quality of Connellsville coke always stood pre-eminent. An indication of how highly it was regarded may be gained from a statement made by R. W. Raymond, President of the American Institute of Mining Engineers, at a meeting of the Institute held in Pittsburgh in October 1872. "I saw in the neighborhood of Salt Lake City, Utah, last summer several carloads of Connellsville coke costing, when delivered, about $33.00 per ton and that price was more economical than charcoal as a smelting fuel."

Even as late as 1915 Connellsville coke was considered the standard coke of the country, its quality being the yardstick against which cokes from all other areas were judged. As far as the other districts of the Appalachian region were concerned, excellent beehive coke was also made in Virginia, West Virginia, and from some of the Alabama coals. The cokes of Kentucky, Ohio, Tennessee, and Georgia were less highly rated.

A characteristic feature of beehive coking is, of course, the production inside the oven of the heat necessary for carbonization. The earlier ovens were made of masonry, firebrick, and tile, and were simple, dome-shaped structures about 12 ft in diam and 7 or 8 ft in height. The floor was constructed of flat tiles, and the domed roof provided with a charging port which also served as a vent for the combustion gases. An arched door built into the side of the oven enabled the air for combustion to be admitted, and also allowed leveling of the coal charge and quenching and withdrawal of the coke after carbonization had been completed. The ovens were built in single rows or banks, and in double rows with ovens back to back or in an interlocking or staggered formation.

The first step in preparing for beehive operations was to start a fire of wood and coal in the oven, the side door being partially bricked up so that only adequate air for combustion was allowed to enter. When the oven reached temperature, the side door brickwork was removed, the chamber cleaned, and the door re-bricked again, a suitably-sized hole for leveling of the coal charge being left. Five or six tons of coal were then charged through the roof and leveled off to a depth of some 2 ft. Under the action of the heat stored in the oven walls, volatile evolution occurred, and combustion took place mainly above the coal charge. Coking proceeded downwards, and the carbonization time varied between 48 and 72 hrs.

Control over the quantity of air admitted to the oven was of considerable importance in beehive operations, and the area of the opening in the side door through which the air entered had to be regulated throughout the coking period if the best results were to be obtained. From a maximum at the time of greatest volatile evolution, the air supply



Cross-sectional view of beehive coke ovens.

was decreased as coking proceeded and the liberation of combustible gases diminished. During the latter stages of the carbonization process, considerable restriction of the air supply was necessary if severe losses due to combustion of the coke were to be avoided.

After the completion of coking, the charge was quenched in the oven and withdrawn for the next cycle to be commenced.

Naturally, the cost of making coke in the beehive oven varied according to the local conditions in any particular producing area. A cost of $1.6735 per ton has been quoted as applying to Connellsville beehive ovens in the year 1899.

As with any other industry, the early days of beehive coking were characterized by simple structures and crude methods. Charging and leveling of the coal were carried out manually, as was raking out of the coke; air regulation was obtained by the rough-and-ready method of inserting or removing bricks in the side door as appropriate; and of course no attempt was made to recover the sensible heat of the waste gases or the heat of combustion of the unburned gaseous or liquid decomposition products. As time went on, however, more sophisticated methods of oven building, operation, and materials handling emerged. Shaped firebrick for the charging ports, domes, and doors came into use, as did the adoption of silica for the roof. Annular air admission passages were provided on some ovens, perforations being used in an effort to obtain equal air distribution above the level of the charge. It was claimed that such an arrangement gave an increased coke yield. Machinery for withdrawing the coke was devised. An English machine patented in 1891 by Thomas Smith of the Thorncliff Iron Works, Sheffield, was put into use near Latrobe, Pa., where in 1895 the Latrobe Coal and Coke Co. started up a plant of 30 Newton Chambers beehive ovens. The Smith coke-drawer consisted of two trucks coupled together and running on a track parallel to the ovens. One truck carried the extractor itself, while its companion carried a small, upright boiler of 9 hp which supplied steam for propelling the trucks and operating the coke-puller. The extractor consisted of a steam engine by means of which a wedge-shaped shovel was pushed under the coke and then withdrawn bringing the coke with it. The machine was said to be capable of drawing four ovens per hour at about one-third the cost of hand labor. The Newton Chambers beehive ovens at Latrobe were also notable for the fact that they were built for by-product recovery. After a short period, however, attempts to recover gas, ammonia compounds, and tar were abandoned.

Another mechanical extractor, the Hebb coke-drawer invented by Mr. J. A. Hebb of Hopwood, Pa., utilized a hoe on a rack which was actuated by a motor-driven pinion. Mounted on rails in front of the oven doors, the machine was equipped with an integral turntable which allowed the hoe to reach all parts of the oven floor. Because of the circular shape of beehive ovens, coke-pullers were only partially successful, and drawing had to be finished off by hand.

Efforts to utilize waste heat for steam generation were made. At the Pratt mines of the Tennessee Coal, Iron, and Railroad Co., two waste heat boiler plants were installed, each of which took the heat from its individual battery of 12-ft bank beehive ovens. Each steam-raising plant consisted of two batteries of 46 in. x 26 ft boilers. The waste gases from the ovens were delivered by short 12-in. diam connecting flues into a 3½-ft diam main flue which was built in contact with the rear walls of the ovens. It is stated that the quantity of coal required for steam raising at the mines was reduced from 1500 to 300 tons per month, and that the savings which resulted from the adoption of waste heat recovery were $18,000 per year.

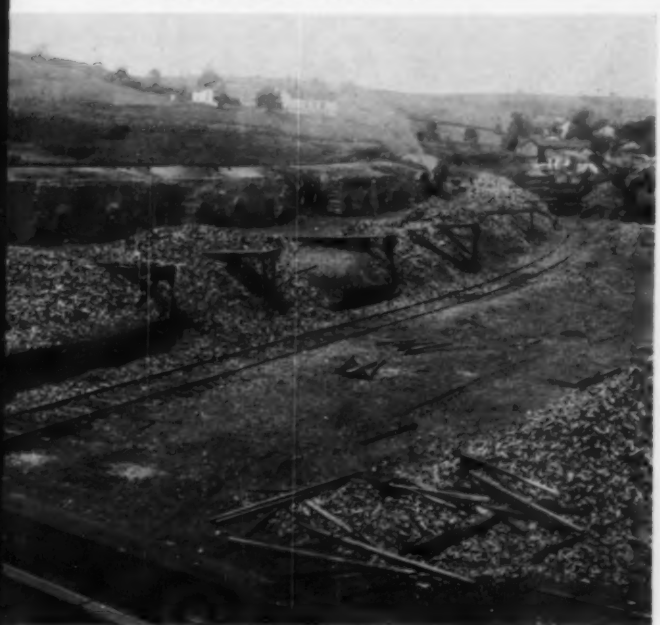
Another method of waste heat utilization was that exemplified by the Ramsay Patent Beehive Oven in which the decomposition products were taken off from the top of the oven, traveled down through external, vertical flues, and on into horizontal combustion flues located below the hearth before being vented through individual stacks.

Adaptations of the beehive oven to allow for mechanical withdrawal of the coke appeared in the old Welsh drag-oven, and later in the Thomas oven. The former was built as an arched chamber 12 ft in length, 7 ft in breadth, and 6 ft high. One end of the oven was walled up and had a flue for gas discharge. The opposite or front end was provided with

doors through which the coke was taken out by means of an iron drag-bar which was positioned on the oven floor before charging with coal. At the end of the coking cycle an engine-driven winch pulled the drag-bar across the floor, thus removing the whole coke mass which was then quenched outside the oven. The Thomas oven was very similar to the Welsh oven but was considerably longer and had doors at both ends. It was thus unnecessary to place the drag in position before charging. As with the Welsh oven, external quenching was practiced. In both cases the principle was that of beehive coking, and in neither quality nor yield did the product differ greatly from that obtained from the older ovens. The *longitudinal oven*, on the other hand, which was yet another modification of the beehive, was claimed to give an increased yield and better coke at a lower operating cost. Originally developed in England, ovens of this type allowed complete mechanization of the modified beehive oven to be attained. Charging was carried out from larry cars running on tracks above the ovens, and the coal was mechanically leveled. Discharging was done by means of a mechanical pusher which delivered the coke to conveyors and then into coke cars.

Excellent metallurgical coke was made in the beehive oven, and for many years after the introduction of by-product ovens, opinion, in England and America especially, was that only in beehive installations could a really first-class coke be produced. Because of its operating characteristics, however, the beehive oven was somewhat selective in the type of coal that could be used, and could not successfully be employed to carbonize the less strongly coking coals. Comparatively low initial oven temperatures, together with the fact that the charge is, in effect, heated from the top only, combine to give a slow rate of heat penetration, and accordingly much of the charge is at a low temperature for a considerable period. While this slow initial heating makes for the formation of a well developed cell structure, it also makes mandatory the use of a coal with strong coking properties. Beehive coke is dense and hard with a silvery, glossy sheen. It is produced in large, rather long pieces. A typical analysis of coke produced from Pittsburgh seam coal in the Connellsville area at the turn of the century was: Moisture—0.5 pct; Fixed Carbon—87.5 pct; Ash—11.3 pct; Sulfur—0.7 pct; and Phosphorus—0.03 pct.

Beehive coke ovens between Greensburg and Mount Pleasant in the Connellsville District of Pennsylvania.



Although coke yields of up to 65 pct from beehive ovens were quoted in the literature of the day, not all authorities agreed that such efficiencies were being generally attained. One of the most trenchant critics of the way in which beehive ovens were being operated was A. W. Belden of the Bureau of Mines (*Metallurgical Coke*, US Bureau of Mines Technical Paper No. 50, 1913). Belden considered that the process of coking had not greatly altered since the evolution of the beehive oven began with the ovens of McCormick, Campbell, and Taylor in 1841, and attributed the higher efficiencies of later years mainly to improvements in the ovens and better coal preparation. "To this day", wrote Belden, "the burning of coke, except in a few instances, is left in the hands of unskilled laborers, and technical knowledge of coking processes is woefully lacking. Until 1896, when the by-product coking industry began to grow appreciably, little was heard of any technical study or deep thought being applied to coke making." After commenting that, "The determination of the coking properties of any coal was generally decided by the report of practical coke burners from the Connellsville region", Belden tartly reminded his readers that, "The very fact that Connellsville coal produces coke of excellent quality no matter how inefficiently the ovens are handled, should preclude the use of such evidence." Skeptical of the percentage coke yields which were being reported at the time, Belden noted that, "The handling of the modern beehive oven, as practised in all parts of the country, undoubtedly gives a lower yield of coke than might be obtained, but the matter of increased yield, even when brought to the attention of those in authority, is most often passed over with the remark, 'We are doing well enough and making money, so why should we make any change?' One reason why it is so hard to impress the importance of greater efficiency on the mind of the coke manufacturer is that he does not know what yield he is getting. He is satisfied to use figures that show the amount of coke produced if they come anywhere near what he thinks he ought to obtain. The 1911 returns to the Division of Mineral Statistics of the United States Geological Survey show the average yield of coke in beehive ovens for the country to be 64.7 pct, but if figures showing the actual tonnages of coal charged and coke produced were obtainable, the yield would probably more nearly approach that in mounds or piles, namely 59 pct. There is no doubt that with proper supervision of the burning of coke this figure could be increased 3 to 5 pct. A conservative estimate of the direct loss from the 27,703,644 tons of coke produced in this country in bee-hive ovens in 1911 is 1,154,318 tons worth over $3,266,350. The gratifying increase in by-product coke production, the output being 22.07 pct of the total production in 1911, shows that the country is waking up to the necessity of curtailing this enormous waste. . . . The fact that the country's best coking coals are rapidly becoming

exhausted makes the scientific study of the process of coking more imperative, and it is only the by-product oven that can give the proper answer to the many questions asked."

There can be little doubt that Belden's strictures were well merited by large sections of the beehive industry. Then, as now, human inertia and resistance to the adoption of sound technological principles allowed rule-of-thumb methods to persist long after these had been proved inadequate. Admittedly, the mechanism of the coking process was a subject about which almost nothing was known, and the industry had been forced to depend on the application of practical skills rather than of scientific knowledge. There were, however, no mysteries about the need for and benefit from such ordinary practices as proper leveling of the coal charge, or careful air regulation over the whole carbonization period. Yet all too often such matters were neglected or ignored to the detriment of both coke quality and yield.

It is likely that beehive oven managers often had trouble in maintaining good operating procedures because of the nature of the work involved. The operation of beehive ovens was hot, dirty, and arduous, especially before the introduction of mechanical methods of leveling, or coke drawing. Only unskilled labor was likely to be attracted to such tasks, and intensive supervision must have been required to obtain consistently good results. The immediate supervisor of the labor force was the foreman or *yard boss*, who was responsible to the superintendent for ensuring that the plant was properly run. "He must keep a record of each oven drawn and charged each day. He must see that the charges are not too large or too small for the 48-hour or the 72-hour coking periods, and that no ovens are drawn until the coke in them is properly coked. The drawers, chargers, levelers, daubers, ash boys, and laborers are under his direct charge, and he must see that they report for duty at the time specified for the days run to begin and that there are no unnecessary delays about a coke plant, as they occasion expenses and loss of coke by retarding the charging of the ovens beyond the regular time for quitting work for the day.

"The coke boss must further see that neither too much nor too little water is used in watering the coke, that the supply of railroad cars arrives at the plant at the proper time, that the coke is loaded properly, that the cars are properly carded for shipment, the yards kept clean, that the water supply is at all times in good shape, and that the ovens receive the necessary repairs, as they deteriorate from constant use. He must also watch the entire plant to see that at all times it is kept in first-class working shape, and that all persons employed at the plant do their work properly and at the right time, as the matter of having certain work done at certain times, and on certain days, is a very important matter connected with the successful manufacture of coke."

A good foreman had to be especially watchful for what a contemporary account refers to as *coke drawers' tricks*. To the hot and toilsome task of pulling coke there were evidently those who applied a measure of cunning as well as a lot of brawn. Thus, "Men who pull coke from the ovens are paid so much per oven and not by tonnage; hence, it is an object to them to have as little coke as possible to draw. Each man is given a block of six ovens; thus he will have three ovens daily to pull. When one oven is being watered down, another oven, if it has the bricks of the door pulled down, can have its coke burning up, so that when it comes time to water the oven a large quantity of coke has been consumed and there is not so much to draw. During the time the coke has been thus burning the oven has also been cooling off; and when it has been watered and left standing without proper attention it is further cooled off with the result that poor coke will be had from the next drawing. To hasten the coking of the coal in an oven, an occasional brick may be knocked out from the door; the increased quantity of air admitted by this means will cut the coke out very fast, and as it is difficult to chink up such holes the ovens must be banked as soon as coking is finished, for otherwise it will cool as well as burn up coke."

Manifestly, the job of yard boss was no sinecure. Indeed, good foremen were, as they still remain, the very backbone of any enterprise. Yet one sees few testimonials to the loyalty and skill of such men, who, standing between management and organized labor, often neglected by one and suspected by the other, contributed much to their company and industry.

The last two decades of the nineteenth century were marked by a steady expansion of the beehive coking industry. By the year 1900 almost 20 million tons of coke were being produced from 57,399 ovens.

In 1910, there were 100,362 beehive ovens in existence, from which 34,570,076 net tons of coke were produced, but the threat of the seventeen-year-old by-product industry was becoming very evident. As the twentieth century opened, some 5 pct of the total coke made in the United States came from by-product oven; by 1910 the percentage had increased to 17.1.

The heyday of beehive coking lay between 1900 and 1920. In only two of these years (1900, 1919) was production less than 20 million tons, and in 1916 an all-time record was reached when 35,464,224 tons of beehive coke out of a national total of 54,-533, 585 tons were made. After the first World War, however, the beehive industry lost its position as the major producer of metallurgical coke. Since that time it has been a marginal source of supply, useful in times of national emergency but contributing in only a minor degree toward the demands of blast furnace or foundry.

(To be continued)

Table III. Beehive Coking During the Last Two Decades of the 19th Century

Year	No. of Ovens	Coke Production, Net Tons
1880	12,372	3,338,300
1881	14,119	4,113,760
1882	16,356	4,793,321
1883	18,304	5,464,721
1884	19,557	4,873,805
1885	20,116	5,106,696
1886	22,597	6,845,369
1887	26,001	7,611,705
1888	30,059	8,540,030
1889	34,165	10,258,022
1890	37,156	11,508,021
1891	40,057	10,322,688
1892	42,002	12,010,829
1893	44,189	9,464,730
1894	44,760	9,187,132
1895	45,493	13,315,183
1896	46,784	11,705,735
1897	47,358	13,027,072
1898	47,883	15,752,764
1899	48,583	19,762,035
1900	57,399	19,457,621

Grain Refinement by Vibration

by J. L. Walker, General Electric Co.

The use of vibration as a tool to control structures produced during solidification of ingots and castings has been the subject of considerable discussion recently. Although it does not appear that vibration can solve all of the grain size and shape problems encountered in ingot and foundry practice, it does provide the metallurgist with an additional means of grain size control. It has been reported by many investigators that vibration of the proper intensity transmitted either to the mold or introduced directly in the melt will produce grain refinement in a variety of alloys. Although the technique has been known and used for some time, there is still considerable diversity of opinion regarding the specific mechanism or mechanisms by which the introduction of sonic energy into a melt can increase the number of grains that form during solidification.

Some of the earlier workers tended to use the ultrasonic frequencies to produce structural changes in metals undergoing solidification. However, the work of Hinchliff and Jones[1] indicated that effective grain refinement could be produced in a high-temperature alloy using subsonic frequencies. They also reported that increasing frequency rather than increasing amplitude had the most potent effect on grain size. Richards and Rostoker[2] showed in an investigation of the effect of vibration on the solidification of an aluminum alloy that a plot of grain size vs displacement yields a frequency independent correlation. These somewhat conflicting results on different alloy systems amplify the need for a more fundamental understanding of the mechanism by which vibration effects solidification.

One of the earlier mechanisms proposed assumed that cavitation occurring in a melt under the influence of intense ultrasonic irradiation shattered dendrites producing showers of fresh nuclei for the formation of new crystals[3]. Direct observation of the crystallization of NH_4Cl has indicated that the fragmentation hypothesis is unable to account for the grain refinement observed during vibration at sonic frequencies[4].

A more generally accepted mechanism by which vibration can produce grain refinement has been described by Richards and Rostoker[2]. In this description it is hypothesized that vibration directly increases the nucleation rate. This increase in rate occurs presumably by the effect of pressure fluctuations on the size of the critical nucleus from which crystals grow. This mechanism has been discussed in some detail by Freedman and Wallace[5] who point out that since the solid is usually more dense than the liquid, the LeChatelier principle would predict that the pressure wave of vibration would tend to promote solid state; i.e., increase the effective rate of nucleation. The magnitude of the pressure required to promote homogeneous nucleation at a sensible rate can be calculated by using the Clausius equation. It is assumed in the calculation that the effect of pressure is equivalent to that of lowering the temperature by an amount required to produce the supercooling at which nucleation will occur. One important conclusion drawn as a result of this kind of calculation is that either nucleation is heterogeneous or the pressure fluctuations produced by vibration are very large in magnitude; i.e., tens of thousands of atmospheres. Since pressures of this magnitude are not generated directly by the kinds of equipment used in vibration experiments, it follows that either vibration causes catastrophic cavitation which gives rise to very high pressures or nucleation occurs on catalysts accidentally included in the melt.

In general the grain size of a casting is determined by the thermal gradients during solidification; by the amount of alloying constituents available to promote constitutional super-cooling; and by the number, kind and distribution of potential nucleation catalysts available to promote nucleation. Any general hypothesis that rationalizes the effect of vibration on cast structure must consider its effect on all of these variables. For example, it might be expected that intense vibration will have a significant effect on the superheat and temperature gradients in the melt during solidification. Lane, Cunningham and Tiller[6] have discussed the effect of ultrasonic radiation on solute distribution, temperature distribution, and nucleation frequency. They pointed out that any change in solute distribution could arise only through convective mixing of the liquid adjacent to the interface. Since convective mixing tends to reduce the magnitude of the constitutional supercooling the effect of vibration on constitutional supercooling is to reduce the probability of equiaxed grain formation. In considering the effect of ultrasonic radiation on the thermal gradient at a freezing interface, the authors pointed out that it is possible that the heat generated as a result of attenuation at the interface may be of sufficient magnitude to inhibit freezing while the superheat flows out through the interface. The thermal conditions in the casting during solidification would then be similar to those in castings poured with very little superheat, a condition that generally promotes grain refinement. The amount of acoustic energy required to produce the growth inhibiting effect is shown to be considerably higher than that required to produce grain refinement. Since it is not necessary to propose an effect of vibration on either solute or temperature gradients to explain their observations, Lane, Cunningham and Tiller concluded that the important effect of vibration involves nucleation.

A quantitative explanation of the precise mechanism by which vibration of a melt induces nucleation will depend upon the results of theoretical and experimental work now underway. This work is directed toward determining the effect of pressure and turbulence on nucleation kinetics.

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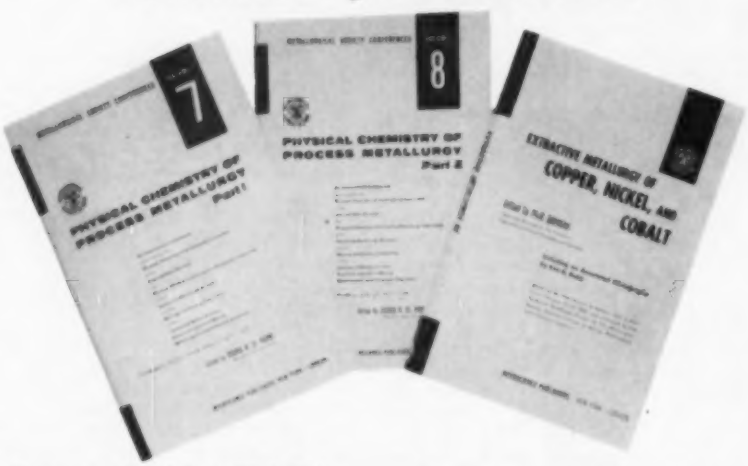
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PHYSICAL CHEMISTRY OF PROCESS METALLURGY Part I
8
PHYSICAL CHEMISTRY OF PROCESS METALLURGY Part II
EXTRACTIVE METALLURGY OF COPPER, NICKEL, AND COBALT

Response of Metals to High Velocity Deformation will be the title of Volume 9 in The Metallurgical Society Conference series published by Interscience Publishers, Inc. This volume contains the proceedings of a conference sponsored by IMD's Physical Metallurgy Committee at Estes Park, Colo., on July 11-12, 1960.

A grant from the National Science Foundation made it possible to bring two speakers from England and to assist in bringing conferees who had no other source of travel funds.

The purpose of the conference was three-fold—first, to compare notes on experiments already performed but not published, second, to try to pin down the major problems which are impeding understanding of the effects of deformation, and third, to educate a considerable number of amateurs who were anxious to obtain a beginning knowledge of the subject.

The book is divided into two sections, the first concerned with *High Velocity Deformation*, and the second with *Shock Phenomena in Metals*.

Two other volumes (7 and 8) in the conference series were recently published by Interscience. These volumes entitled **Physical Chemistry of Process Metallurgy** contain the proceedings of a conference held in Pittsburgh, April 27-30, 1959. Part I (Volume 7) contains 35 papers, and the section titles include *Physical Chemistry of Metallurgical Phases*, *Thermodynamics of Metals*, and *The Nature and Structure of Liquid Metals*. This volume contains approximately 650 pages and sells for about $23.00. The special AIME members' price is about $18.40.

Part II (Volume 8) contains 32 papers. Included in the section titles are *Solidification of Metals*, *Desulfurization in the Steel Plant*, and *Hydro- and Electro-Metallurgy*. This volume also sells for about $23.00, with a special AIME members' price of about $18.40.

Extractive Metallurgy of Copper, Nickel, and Cobalt contains the proceedings of a symposium held in New York February 15-18, 1960, and sponsored by the Extractive Metallurgy Division. Edited by Paul Queneau (see Editorial, page 343), the book contains approximately 650 pages, and sells for about $23.00. The special AIME members' price is about $18.40 Some of the session titles are *Fuel-Fired Smelting and Converting*, *Electric Furnace Smelting*, *Atmospheric and Elevated Pressure Leaching*, and *Refining*.

Plans Announced For U.S. Fall Meeting of British Metals Group

The Iron and Steel Institute (of Great Britain) has accepted the invitation of The Metallurgical Society of AIME to hold a special meeting in the United States and Canada October 17-November 8. The group has also been invited by the American Society for Metals to attend their Annual Congress and Metals Exposition in Detroit.

Dr. James B. Austin, vice president of research and technology, U. S. Steel Corp., is chairman of the Organizing Committee. The itinerary, divided into sections, follows:

Section I: The visitors will arrive in New York on October 17 or 18, and attend the opening reception and social functions on October 19. An all-day visit will be made to the Fairless Works of U.S. Steel Corp. on October 20, and the group will leave by special train for Niagara Falls on Saturday, October 21.

Section II: This part of the trip will begin on Sunday with sightseeing at Niagara Falls. Monday through Wednesday (Oct. 23-25) will be devoted to visits to three or more of the following plants: Atlas Steels Ltd., Welland, Ont.; Dominion Foundry & Steel Co. Ltd.; Hamilton, Ont.; Steel Co. of Canada Ltd., Hamilton, Ont.; Union Carbide's Linde Co. Laboratories; and Union Carbide's Metals Research Laboratories.

Section III: The group will leave Niagara on Wednesday and travel to Cleveland, where Thursday will be devoted to an all-day visit to the Cleveland Works and Research Center of Republic Steel Corp. Scheduled for Friday is a trip by bus to the Massillon and Canton Works of Republic Steel, proceeding in the late afternoon to Pittsburgh. Members not wishing to visit Massillon and Canton, may travel directly from Cleveland to Pittsburgh.

An alternate arrangement will permit those who wish to visit Detroit rather than Cleveland. This group will travel by train from Niagara (Welland) to Detroit on Wednesday. On Thursday they will attend the American Society for Met-

als Congress, Metals Exposition and Banquet. On Friday (Oct. 27), the visitors will fly to Akron, then travel by bus to Massillon and Canton. The Ladies Program for the day offers either an all day visit to an automobile plant in Detroit, with a plane trip to Pittsburgh in the evening, or an all-day bus trip from Detroit to Pittsburgh. Institute members who do not wish to visit Massillon and Canton may join the ladies program.

Section IV: In Pittsburgh, Saturday through Wednesday (Nov. 1) will be devoted to visits to at least three of the following: U. S. Steel Corp.'s Homestead Works and Monroeville Research Laboratories; Jones & Laughlin Steel Corp.'s Aliquippa Works; Carnegie Institute of Technology; and National Forge Co. at Warren, Pa. The trip to the National Forge Co. will be made by plane on Wednesday; members not wishing to fly will not be able to make the trip.

Section V: On Thursday, (Nov. 2) the group will go from Pittsburgh to Washington. They will have a choice of flying to Washington in the afternoon after a free morning in Pittsburgh; flying to Washington and having a free afternoon there, or leaving Pittsburgh by bus in midmorning, having lunch en route to Washington.

Friday will be devoted to an all-day visit to the Sparrows Point Works of Bethlehem Steel Corp. On Saturday there will be a short bus tour of Virginia, and after lunch, an afternoon visit to Mount Vernon. No activities have been planned for Sunday morning, (Nov. 5). Sunday afternoon will feature visits to the Lincoln and Jefferson Memorials, Arlington Cemetery, and Washington Monument. The Group will leave Washington on Monday morning.

Section VI: After leaving Washington by bus, the group will have lunch in Philadelphia and make a sightseeing tour in Philadelphia; then travel by bus to Princeton, and after a one hour stop, proceed to New York, arriving about 8 pm. Those who wish may travel by rail or air directly from Washington to New York. The Institute members will have Tuesday free in New York. The group is scheduled to leave for England on Wednesday, November 8.

Extension I: An alternative excursion is being arranged for members who wish to see more of the major iron and steel works than have been included in the main program. Ladies will be able to travel on this trip, but will not be able to visit the works. This group will leave Washington by plane on Sunday, flying to Cincinnati. On Monday they will visit the works and research laboratories of Armco Steel Corp. at Middletown, Ohio; flying to Chicago that evening. Tuesday (Nov. 7) through Thursday will be devoted to visits in and near Chicago to the following plants: U.S. Steel Corp.'s Gary Steel Works and Furnaces, Gary Sheet and Tin Mills, and the South Works, Chicago; and Inland Steel Co.'s Indiana Harbor Works. On Thursday or Friday the visitors will leave Chicago by train or air for New York, or directly by air for Europe.

Extension II: Thomas Cook & Son Ltd., will make arrangements for members to visit Colonial Williamsburg and Richmond after the Washington visit.

All members of the British Institute and their wives, including those resident in the U.S. and Canada, may register for the whole meeting or for one or more complete sections of the meeting. Registrations will not be accepted for individual visits or excursions or functions which do not comprise a whole section of the meeting.

Registration forms may be obtained from the Secretary, The Iron and Steel Institute, 4 Grosvenor Gardens, London, S.W.1.

Basic Metals Industry Will Be Subject Of N. E. Regional Conf.

General Electric Research Laboratory, Schenectady, N. Y., will be the scene of the Fifteenth New England Regional Conference May 18-19. Sponsored by the Boston, Connecticut, and Hudson-Mohawk sections, AIME, the conference will have the theme, *The Basic Metals Industry—Technical and Economic Perspectives.* Three technical sessions on *How It Grew, What It Is,* and *Where It's Going* will be presented.

Speakers from the aluminum, copper, and steel industries will take part in each session. J. S. Smart, Jr., President of The Metallurgical Society and general sales manager of American Smelting & Refining Co., will discuss the copper industry and where it stands today.

Other speakers, representing various facets of the industries, include C. A. Moore, Copper Products Development Assn.; L. P. Favorite, Aluminum Co. of America; Wm. V. Lidgewood, Aluminium Limited Sales Inc.; LaVerne Eastwood, Olin Mathieson Chemical Corp.; R. D. Woodward, Bethlehem Steel Co.; Rev. Wm. T. Hogan, S.J., Fordham University; and T. T. Magel, Allegheny Ludlum Steel Corp. Panel discussions after the second and third sessions will include other representatives of the basic metals industries.

The conference will conclude Friday afternoon, May 19, with a visit to the Watervliet Arsenal, Watervliet, N. Y. Registration information is available from Prof. David A. Thomas; Metallurgy Dept., Room 16-502; Mass. Institute of Technology; Cambridge 39, Mass.; Telephone UN 4-6900, Ext. 3322.

HOWARD BIERS

Howard Biers Named Honorary Officer Of Iron & Steel Institute

The election of Howard Biers, of New York, internationally distinguished metallurgist and consulting engineer, as an honorary vice president of The Iron and Steel Institute, London, has been announced in New York coincidentally with his designation by AIME as its representative at the Annual General Meeting of the Brtish organization May 3-4.

Dr. Biers, who is an Honorary Life Member of AIME, is a consultant to Union Carbide International Co. He has been an honorary member of the British organization since 1955. In that year the British Institute also named Ernest O. Kirkendall, Secretary of AIME, and the late William E. Eisenman as Honorary Members.

The recipient of many awards here and abroad and an honored member of many professional organizations, Dr. Biers was informed by The Iron and Steel Institute of Britain that his election as Honorary Vice President, "a somewhat exceptional distinction", was an expression of appreciation of his activities in its behalf through the years. He is the first American to hold the position since the death, in 1943, of James Farrell, president of U.S. Steel Corp.

There are more than 400 American members of the British Institute.

Dr. Biers, who lives in Norwalk, Conn., was born in New York City. His degrees include Bachelor of Science from the University of Virginia, Master of Science from Massachusetts Institute of Technology, and Doctor of Mining and Metallurgical Science from the University of Mining and Metallurgy in Austria (the only American ever to receive the last named degree from this university).

A member of AIME since 1953, Dr. Biers is also a member of many other technical and professional groups in this country and abroad.

News of The Metallurgical Society of AIME

Some Actions by Society Board of Directors (Meeting of March 1, 1961)

"Metallurgy" — Association With "Engineering"

Concern was expressed by the Committee on the Metallurgical Profession with the increasing tendency to dissociate the profession of metallurgy from engineering. The following resolution was adopted:

WHEREAS the Board of Directors of The Metallurgical Society of AIME notes with concern an increasing tendency to dissociate the metallurgical profession from engineering,

BE IT THEREFORE RESOLVED that in publicity and in other public pronouncements, the activities of The Metallurgical Society of AIME be associated with both metallurgy and with metallurgical engineering. This resolution was seconded and carried.

Untimely Death of C. T. Evans, Jr.

The untimely death of C. T. Evans, Jr., a member of the Committee on the Metallurgical Profession, and also a member of the Education Committee, was recognized by the following resolution:

10. *Report of the Committee on the Metallurgical Profession:*

W. R. Hibbard, Jr., reported on the untimely death of C. T. Evans, Jr., a member of the Committee on the Metallurgical Profession, and also a member on the Education Committee. Dr. Hibbard proposed the following resolution:

WHEREAS the Board of Directors of The Metallurgical Society notes with sorrow the death of C. T. Evans, Jr., an active member who served both on the Education Committee and the Committee on the Metallurgical Profession,

BE IT THEREFORE RESOLVED that the Board of Directors of The Metallurgical Society wishes to express its sympathy to Mrs. C. T. Evans, Jr., and to the Universal-Cyclops Steel Corporation on behalf of The Metallurgical Society and its membership.

Closer Relations With AIME President

Carleton C. Long has stated that "during my term of office as President of The Metallurgical Society, I have been fortunate enough to have met with a wide geographical distribution of Society and Institute members. In the course of my travels, I have become aware of a tendency to forget that in back of all of the technical activities in which a member finds his interest of the moment there is a coordinating body—the AIME. I have also observed how effective in 'bringing the Institute to the members' an energetic, time-available person like Joe Gillson can be. Not all Presidents of the Institute will have the time or the energy of a Joe Gillson to take the initiative in meeting with specialized groups. Almost any Institute President, however, will be willing to extend himself to any reasonable degree if he feels he is genuinely wanted and welcomed by a particular group."

Accordingly, a resolution, sent to Society Directors by letter ballot on January 12, was subsequently approved:

WHEREAS with the consummation of the recommendations of the Long Range Planning Committee, the Institute was reorganized into three operating Societies, and

WHEREAS the wisdom of this recommendation has demonstrated itself in a vigorous growth of the Societies and a healthy proliferation of technical committee activities and other services to members, and

WHEREAS the increasing magnitude of these professional activities has tended to veil the unifying function of the Institute, and

WHEREAS an effective means for communicating between the Institute and its constituent Societies is a personal appearance of the Institute President at official Society functions, now

BE IT THEREFORE RESOLVED that the Metallurgical Board recommend to the governing bodies of the other Societies and of the Institute that the President (or President-Elect) of the Institute be invited officially to attend such functions as Meetings, Dinners, Lectures, Conferences, and other gatherings where an opportunity exists for a significant number of members to meet or hear the President of the Institute, and

BE IT FURTHER RESOLVED that this Resolution be communicated to the governing bodies of the other Societies, of the Institute, and of the Local Sections.

Additions to Administrative Rules

The following addition to the "Administrative Rules for the Interpretation and Operation of the Bylaws of The Metallurgical Society of AIME" were submitted to the Board of Directors of The Metallurgical Society on January 12, 1961, and subsequently approved:

Proposed

Additions to "Administrative Rules for the Interpretation and Operation of the Bylaws of The Metallurgical Society of AIME"

Under Article VI, add new Section 2, Rule 1:

"The annual Lectures of the Divisions (the Institute of Metals Lec-

ture, the ISD (Howe) Lecture, the EMD Lecture) shall be known collectively as Lectures of The Metallurgical Society. As such, the President of The Metallurgical Society will take responsibility for introducing the Lecturer."

Under Article V, Section 1, Rule 1, add new stipulation "n":

"n. Preside at lectures of The Metallurgical Society, the Institute of Metals Lecture, the ISD Howe Lecture, the EMD Lecture or appoint a distinguished member of the Institute to act in his stead."

Bylaw Changes Announced

The following changes in Bylaws of The Metallurgical Society were submitted to the Board of Directors of The Metallurgical Society on January 12, 1961, and were approved by letter ballot:

A. To Provide for Engineering Management Committee.

Under Article VII, Section 3 ("Standing Committees shall be ....."), add:

j. Engineering Management Committee."

Under Article VII, add new Section 13:

"The Engineering Management Committee shall consist of six members, two appointed each year for a period of three years by the President of the Society with the approval of the Board of Directors. One of the members serving his third year will be designated as Chairman.

a. It shall be the duty of this committee to take cognizance of management activities of use to members, and to seek means of improving the capabilities of members of the Society in this field by providing programs to accomplish these ends, by correlating existing information with respect thereto and by disseminating such information by publication and other means."

These proposed bylaw revisions were approved by the AIME Board of Directors on February 26, 1961.

B. To Provide for Fellow Grade of Member.

These proposed bylaws changes have been tabled for further study by the AIME Board of Directors.

Under Article VII, Section 3 ("Standing Committees shall be ....."), add:

"i. Fellows Nominating Committee."

Under Article VII, add new Section 12:

"The duties and setup of the Fellows Nominating Committee are detailed in Article II, Sections 1 and 2, of these Bylaws."

Under Article II, Section 1, add the phrase underlined:

"Any member of AIME in good standing may become a member of the same grade of the Society by indicating his choice in writing to AIME headquarters. . . ."

Under Article II, add new Section 2:

"Fellows shall be persons who have been full members of AIME for at least five years continuously and who shall prove qualified for elevation to the grade of Fellow by reason of outstanding contributions to the metallurgical arts and sciences. They shall be elected by the Board of Directors in the manner set forth in these Bylaws. Fellows shall be entitled to use the title, 'Fellow of The Metallurgical Society.'

a. A two-thirds vote of the Board of Directors shall be required for elevation to the grade of Fellow after consideration of the following specific requirements:

1) A candidate must have a good personal reputation and have won recognition for outstanding or notable contributions in any phase of metallurgy. He must have attained distinction as an eminent authority in some aspect of the broad field of metallurgy as set forth in the following Section 2, a, (1), (a). Outstanding service to The Metallurgical Society shall be considered in rating nominees who are otherwise qualified."

a) Broad and productive scholarship in production, manufacturing, engineering applications, science, teaching or management as evidenced by (i) the publication of articles and books of merit on metallurgical work supervised or done by the nominee, (ii) the granting of patents to the nominee on scientific and technical developments in materials, process methods or equipment in various fields of metallurgy, (iii) direction of important research or engineering work, and (iv) responsibility through management for nationally known metallurgical improvements and developments.

b. No more than five Fellows will be elected each year, and the maximum number of living Fellows shall not exceed one hundred (100).

c. The method of nomination of candidates shall be as follows:

1) Nominations for elevation to the grade of Fellow shall be made on forms approved by the Board of Directors.

2) The Fellows Nominating Committee shall consist of five (5) Fellows, one appointed each year for a period of five years by the incoming President of the Society, and the three (3) most recent living past presidents of The Metallurgical Society. The appointed member senior in service shall be chairman.

a) The President of The Metallurgical Society shall fill any vacancy that occurs during the year.

b) Retiring members shall not be eligible for reappointment until a period of three (3) years has elapsed.

d. Any member of the Fellows Nominating Committee or any five members of The Metallurgical Society may propose a member's name to the Nominating Committee on forms as provided for in Section 2, c, (1).

1) The Secretary of The Metallurgical Society shall send to the Chairman of the Fellows Nominating Committee sufficient copies, so that each member will have one, of all properly executed nominations and any supporting statements. The Chairman shall send one copy to each member of the committee.

2) Each member of the committee shall return to the Chairman not later than the first day of August all copies of nominating papers and supporting statements, together with the names of nominees he considers quaified for elevation to Fellow, arranged in the order of his respective preference for them. The Chairman shall select a list of five (5) receiving the highest rating based on the preferential vote of the members of the committee, and transmit the list to the President of the Society not later than the first day of October. Voting to elevate the nominees to Fellow shall take place at the next following meeting of the Board of Directors of The Metallurgical Society.

3) The nominees elected by at least a two-thirds favorable vote of the entire Board of Directors of The Metallur-

(Continued on next page)

Bylaw Changes

(Continued from page 383)

gical Society shall be notified by the Secretary and their acceptance secured. They shall be elevated to the grade of Fellow at the next annual meeting of the Society.
(i) A suitable certificate shall be presented to each Fellow.

4) In the event a nominee is not elected, his nomination will be considered by three consecutive Nominating Committees, after which his papers will be destroyed. A new nomination, however, if presented, will be considered as though it were a nomination presented for the first time.

e. There shall be no special dues for Fellows.
f. The Fellows, with the approval of the Board of Directors of The Metallurgical Society, may establish procedures and rules for the administration of their affairs.
g. In order to initiate the Fellow grade of membership, a maximum of twenty (20) will be elected in 1961. A special Fellows Nominating Committee will be appointed by the President of The Metallurgical Society, subject to approval of the Board of Directors, and its recommendations shall be forwarded to the President of The Metallurgical Society not later than the first day of October. Voting to elevate the nominees to Fellow shall take place at the next following meeting of the Board of Directors of The Metallurgical Society.
l) This section shall be dropped automatically from the Bylaws as soon as the Charter Fellows are nominated and elected.

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Under the auspices of the Four Founder Engineering Societies and affiliated with other renowned Engineering Societies, ESPS offers many years of placement experience in addition to world-wide contacts.
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Roster of Officers and Board Members

THE METALLURGICAL SOCIETY OF AIME

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C. C. Long, Past President
K. L. Fetters, Vice-President
T. D. Jones, Treasurer
R. W. Shearman, Secretary
AIME, 29 West 39th Street
New York 18, N.Y.
D. C. Johnston, Assistant Secretary

Board of Directors: J. H. Jackson, '62, J. H. Hollomon, '62, J. H. Scaff, '63, W. J. Harris, Jr., '64, IMD. Gerhard Derge, '62, R. W. Farley, '62, Michael Tenenbaum, '63, H. B. Emerick, '64, ISD. R. C. Cole, '62, J. D. Sullivan, '62, A. W. Schlechten, '63, A. E. Lee, Jr., '64.

European Competition Discussed At Meeting Of North Ohio NOHC

The Northern Ohio section of the National Open Hearth Steel Committee held its Annual Off-the-Record Meeting in Youngstown March 23, with George McCuskey, vice-president of finance, Youngstown Sheet and Tube Co., as the featured speaker. His topic was *The Challenge of Foreign Competition in Iron and Steel.* He was introduced as the after-dinner speaker by Karl L. Fetters, vice-president of research, Youngstown Sheet and Tube Co., and President-Elect of The Metallurgical Society. Some 360 were in attendance at the one-day meeting at Youngstown's Pick-Ohio Hotel.

Mr. McCuskey related the economic resurgence of Western Europe to problems of the United States' steel industry. Three factors have influenced Europe's post-war development: (1) Replacement of obsolete equipment with up-to-date facilities has resulted in highly efficient operations. (2) Our foreign-aid program completed European recovery much quicker than otherwise and deliberately financed our competition. (3) Europe has achieved a much greater degree of economic unity than anyone would have dreamed possible.

The European Economic Community, created in 1957, is an outgrowth of the European Coal and Steel Community, founded in 1952. With the reduction of tariff barriers among the six participating countries (Belgium, France, Germany, Holland, Italy, and Luxembourg), and the establishment of a common tariff wall, each company has had the incentive to tool up for a larger market. The result has been an increase from 46,000,000 ingot tons of steel produced in 1952 to 80,000,000 ingot tons in 1960. This 74 pct increase compares with an 8 pct increase in the United States.

The speaker warned that unless we recognize our deteriorating international position and take the necessary disciplinary action, we will find that we have had our golden years. We will suffer the gradual loss of our export markets and the growing invasion of our foreign markets. These actions were recommended: (1) Lower production costs per man hour, (2)Encourage more investment, (3) Obtain realistic depreciation allowances, (4) Bring to a halt the inflationary wage increases that are not related to increased productivity.

At the technical session, registrants were welcomed by the Northern Ohio section chairman, Alfred T. Hubbard, superintendent of open hearths, Brier Hill Works, Youngstown Sheet and Tube Co. Serving as session chairmen were J. Schosser, superintendent of open hearths, Roemer Works, Sharon Steel Corp., and E. T. Britt, superintendent of open hearths, Campbell Works, Youngstown Sheet and Tube Co.

First paper on *Mechanical Capping of Steel* was presented by D. M. Snider, metallurgical engineer, Republic Steel Corp., Youngstown. With no change in furnace practice and a little extra care in pouring, a sounder ingot with less segregation can be obtained than with killed or semi-killed steels.

Advantages of a Carbon Sub-Hearth were discussed by John Cleary, practice engineer, open hearths, Republic Steel Corp., Warren, Ohio. It was recognized that the hearth must be of compatible material, of sufficient depth, and be subject to repetitive and constant care if a high level of production is to be maintained. This led to the construction of a hearth of carbon blocks at No. 1 Open Hearth at Warren in 1956.

Experiments with *Oxygen Jets in Youngstown* were described by J. J. McPhillips, practice engineer, Open Hearths, Campbell Works, Youngstown Sheet and Tube Co. (Mr. McPhillips is secretary of the Northern Ohio section, NOHC.)

The technical session was concluded with a panel discussion on *Operation and Maintenance of Basic Roofs.* Participants were R. H. Frushour, general superintendent of Steel Plant and Struthers Plant, Campbell Works, Youngstown Sheet and Tube Co.; R. Oswald, general superintendent, Steel div., Roemer Works, Sharon Steel Corp.; J. E. Smith, superintendent of open hearth and electric melt shop, Republic Steel Corp., and E. L. Wentz, div. superintendent of steel production, U.S. Steel Corp., Youngstown.

(Continued on page 386)

Semiconductors Conf. Slated Aug. 30-Sept. 1 In Los Angeles

The third annual Conference on the Metallurgy of Semiconductor Materials will be held August 30-September 1 at the Ambassador Hotel in Los Angeles. The three day program will be confined principally to *Microelectronic Metallurgical Techniques*, covering substrates, vapor deposition, epitaxy, and thin films, and *Energy Conversion Materials*. A portion of the program will also be devoted to brief reports of *Research in Progress* on topics in the general field of semiconductor metallurgy. A proceedings volume will be published following the conference, but will not include reports of *Research in Progress*. The Southern California section, AIME, will be hosts for the conference.

Chairmen of the principal committees are William V. Wright, general committee and program committee; Craig G. Kirkpatrick, arrangements committee; Fred Burns, attendance and registration committee; James O. McCaldin, finance committee; C. H. Gray, membership committee; and John V. Houston, Jr., publicity committee.

Fall Meeting Abstracts

June 28 is the deadline for receipt of abstracts to be considered for presentation at IMD-sponsored sessions during the 1961 Fall Meeting of The Metallurgical Society, which will be held from October 23-26 in Detroit.

In order to be considered for inclusion in the program, each submission should consist of the following:

1) Three copies of a 200-300 word abstract, typed double-space on one side only. (No photographs, figures, tables, or diagrams, please; however, brief acknowledgments of research sponsorship may be included if applicable.)
2) One copy of an information sheet containing complete mailing address of each author together with title of paper being submitted.

Submissions should be forwarded to:

D. C. Johnston, Secretary, IMD
The Metallurgical Society of AIME
29 West 39th Street
New York 18, New York

Planning the 1961 AIME Semiconductors Conference to be held August 30—September 1 at the Ambassador Hotel in Los Angeles are (l to r), W. V. Wright, general chairman, Fred Burns, J. O. McCaldin, and C. G. Kirkpatrick.

Magnetism Conference Scheduled In Phoenix November 13-16

The Seventh Annual Conference on Magnetism and Magnetic Materials will be held in Phoenix, Ariz., November 13-16, at the Hotel Westward Ho. This conference is sponsored jointly by the American Institute of Electrical Engineers, and the American Institute of Physics, in cooperation with The Metallurgical Society, the Office of Naval Research, and the Institute of Radio Engineers.

Deadline for abstracts is August 18. Further details can be obtained from the local chairman, Peter B. Myers, Motorola Semiconductor Products div., 5005 E. McDowell Road, Phoenix, Ariz.

Metallurgical Society Directory To Be Issued In July

The bi-annual Membership Directory of The Metallurgical Society will be published in July, according to present plans. Inquiries were sent to all Society members in March in order to determine present addresses, company affiliations, and major technical interests. The Directory will include both an alphabetical listing of members' names showing addresses, date of joining AIME, and primary divisional affiliation, and a geographical listing by country, state, city, and company.

A single copy of the 1961 Membership Directory will be forwarded as soon as published to all members of The Metallurgical Society who return the coupon below. This coupon will be used as a mailing label. The Directory will not be included as an insert to JOURNAL OF METALS, as was the case with the most recent Directory, issued in July, 1959. This is the *only* issue in which the coupon will appear.

Please order your copy by returning the coupon *now*. To keep production costs to a minimum, our print order will be based on orders *received by June 1*. A charge of $1.00 per copy will be made for orders received after June 1, or for orders not using the mailing label. Copies can be purchased as long as the supply of copies lasts.

Coupon

(For use by members of The Metallurgical Society of AIME only.)

Please send me one copy of the 1961 Membership Directory of The Metallurgical Society of AIME to be published in July. I certify that I am a member of AIME, and that the Directory will not be used for circularization purposes.

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(Please print legibly. This coupon will be used as a mailing label.)

Joseph Becker Award Is Established By The Metallurgical Society

JOSEPH BECKER

The Metallurgical Society of AIME has established the Joseph Becker Award for distinguished achievement in coal carbonization. The award was created in honor of a famous chemist, inventor, engineer, builder and administrator who, for decades, led and pioneered in many of the advances that made possible this country's eminence in iron and steelmaking.

Mr. Becker is a former president of Koppers Construction Co., forerunner of the Engineering and Construction div. of the Koppers Co. Inc., and is a member of the Koppers Co. Board of Directors. Koppers has provided a fund of $5,000 to establish the award, and The Metallurgical Society hopes to name a winner annually, beginning possibly, in 1962. It will be administered by the Blast Furnace, Coke Oven, and Raw Materials Committee of the Iron and Steel Division.

The Becker Award is "for distinguished contributions in the field of coal carbonization including developments in coal technology that lead to greater efficiency in the use of coke in ironmaking blast furnaces and also improvements in coke-oven design, operation, and coal chemical recovery, resulting in increased productivity or economics."

Conditions of the award require that the winner be a person whose principal interest is in coal carbonization. The award will consist of a gold medal and a certificate of citation.

Holder of 97 patents, Mr. Becker has been one of the major contributors to steelmaking developments in the United States. He is credited with having directed the major portion of a World War I drive to achieve the needed coke-oven production, and in the second World War he directed the engineering and construction of a huge plant for the nation's synthetic rubber program in record time.

He played a major role in launching the steel and gas-utilities industries on a program of heating coke ovens with *lean* gases from blast furnace and producer plants. He also invented the cross-over flue system of heating cross regeneration coke ovens, saving millions of dollars for the industry and producing coke in ovens of much larger capacity. Demonstrating how to coke coals that formerly were regarded unsuitable for coking, he made possible the establishment of steel plants on the west coast.

Born in Essen, Germany in 1887, Mr. Becker went to work as a youth for Heinrich Koppers, famous for improved coke ovens that recovered all the valuable gas tars and chemicals from coke. At 19, Becker became a chemist in the Koppers laboratories.

Becker came to this country in 1910 as a chief chemist of Koppers' operations, later becoming superintendent and pioneering in the chemical recovery coke oven business in this country.

Pittsburgh steel interests purchased control of the Koppers firm in this country in 1914. Mr. Becker, who became an American citizen, was the engineering and construction superintendent in the building of 3,378 coke ovens at 22 plant locations during World War I. In 1922 the Koppers-Becker oven—large, fast, efficient, and economic—took firm hold in the steelmaking industry.

In 1925, Mr. Becker became vice president and general manager and later president of the Koppers Construction Co. In 1950 he became director of the Koppers Co.

In the April issue JOURNAL OF METALS began a series of four articles (for Part Two, see p 000) on the history of coke ovens. Joseph Becker's career and contributions to that history are recounted.

Education

(Continued from page 334)

on both the RCA EMU-2D and Hitachi HU-11 microscopes.

Further information can be obtained from Professor Joseph J. Comer, College of Mineral Industries, Pennsylvania State University, University Park, Pa.

Materials Science

The department of chemical and metallurgical engineering at the University of Cincinnati has announced a new area in which graduate students in the department can major; this new area will be known as materials science. Work leading to both the master of science and doctoral degrees is offered.

The university's new advanced studies are an outgrowth of national needs for new materials of many types. Dr. Michael Hoch, professor of metallurgical engineering, will be chairman of the program.

Students can enter the program from undergraduate backgrounds in either engineering, chemistry, or physics.

North Ohio NOHC

(Continued from page 384)

The technical session was followed by a cocktail hour, courtesy of suppliers, and then the Fellowship Dinner. The invocation was given by the Rev. Arthur C. Joachim, Assistant Minister, First Presbyterian, Youngstown. Serving as toastmaster was Joseph Turner, superintendent, open hearths, Republic Steel Corp., Youngstown. A special guest was Carleton C. Long, Past-President of The Metallurgical Society, and director of research, St. Joseph Lead Co. RWS

EMD Committees Plan Symposia For 1962-63 Annual Meetings

The Extractive Metallurgy Division of The Metallurgical Society has scheduled an International Symposium on the *Extractive Metallurgy of Aluminum* to be held during the AIME 1962 Annual Meeting in New York. R. A. Lewis of Kaiser Aluminum and Chemical Corp. heads the committee in charge of this event. W. R. Opie of Amco Research and Development is program chairman for the New York meeting.

The Long Range Planning Committee of the Division has also scheduled for the Institute's 1963 Annual Meeting in Dallas, a symposium on *Unit Processes in Hydrometallurgy*. This event will be held jointly with the Minerals Beneficiation Division of the Society of Mining Engineers. Prof. Milton E. Wadsworth of the University of Utah is executive chairman of the committee in charge, and F. T. Davis, Colorado School of Mines Research Foundation, is co-chairman.

Books

(Continued from page 342)

An additional paper is included dealing with the application of coulometric determination of tin to the analysis of Zircaloy.

Physical and Mechanical Properties of Columbium and Columbium-Base Alloys, by E. S. Bartlett and J. A. Houck, Battelle Memorial Institute, 62 pp., no price quoted, 1960—Data on seven currently commercial or pilot-production alloys are presented. Of these, the Cb-15W-5Mo1Zr-0.5C-0.05O composition appears to be the most promising for elevated-temperature structural applications, based on strength and recrystallization temperatures. This alloy has a 100-hr rupture stress of 35,000 psi at 2000°F, or 17,000 psi at 2200°F.

Order the report, PB 151082, from the *Office of Technical Services, Dept. of Defense, Washington 25, D. C.*

MEMBERSHIP

Proposed for Membership
Metallurgical Society of AIME

Total AIME members on March 31, 1961, was 35,200; in addition 2,145 Student Members were enrolled.

ADMISSIONS COMMITTEE

W. L. Brytczuk, Chairman; S. C. Carapella, Jr.; J. W. Hanley; T. D. Jones; H. C. Larson; Harold Margolin; Shadburn Marshall.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

Members

Boxall, Douglas G., North Peterborough, Ont., Canada
Casey, John A., Valparaiso, Ind.
Cheney, Donald M., Brigham City, Utah
Cichy, Pawel, Niagara Falls, N. Y.
Clark, Robert, Warren County, Pa.
Davies, Marcus O., Industry, Pa.
Fischbach, David B., Pasadena, Calif.
Fischer, David J., Midland, Mich.
Fleming, L. W., Hobart, Ind.
Foos, Raymond A., Elmore, Ohio
Gardner, Howard R., Richland, Wash.
Gomes, Mario R., Belo Horizonte, Brazil
Haswell, Walter T., Jr., Latrobe, Pa.
Kehl, Nelson H., Martinsburg, W. Va.
Lang, Eugene A., Magna, Utah
Lilley, John N., Copper Cliff, Ont., Canada
Longo, Rocco A., Industry, Pa.
Lothe, Jens, Pittsburgh, Pa.
Magnell, Per A., Stockholm, Sweden
Mends, David N., Sandy Hook, Conn.
Nilson, Wesley G., Bayonne, N. J.
Norwood, Sidney L., Jr., Birmingham, Ala.
Pryor, Michael J., New Haven, Conn.
Schrader, David M., Canton, Ohio
Silvertsen, John M., Minneapolis, Minn.
Simpson, Henry D., Bethlehem, Pa.
Solomon, Kenneth E., Pittsburgh, Pa.
Stupar, Sergei N., Washington, D. C.
Thorp, Randall B., Alabama City, Ala.
Trebing, Robert T., Chicago, Ill.
Truitt, Blair A., Hobart, Ind.
Waitkus, Joseph, Wellsville, N. Y.
Waterhouse, Ernest L., Gary, Ind.
Whitman, Charles I., Bayside, N. Y.
Wilcox, Roger S., Gary, Ind.

Associate Members

Adams, Leon Gadsden, Ala.
Boyce, Huntting L., St. Louis, Mo.
Colacchio, Thomas D., Boston, Mass.
Espersen, Ralph E., El Paso, Tex.
Feil, Chris W., Chicago, Ill.
Geiger, Lewis J., Jr., Hammond, Ind.
Haron, Carl I., Dallas, Tex.
Hartbower, Carl E., Cambridge, Mass.
Jefferson, George K., Gary, Ind.
Lipkowitz, Irving, New York, N. Y.
MacDonald, William H., Gary, Ind.
Moran, William M., Gary, Ind.
Moreland, Jerry C., Omaha, Tex.
Olt, Robert D., East Chicago, Ind.
Roberts, William W., Gary, Ind.
Thomas, Robert P., Sunnyvale, Calif.
Zimmerman, Max D., Gary, Ind.

Junior Members

Barclay, William F., Berea, Ohio
Bell, Roger S., Bedford, Ohio
Berndt, Alan F., Argonne, Ill.
Crocker, Albert J., Philadelphia, Pa.
Dozza, Otto L., Maywood, Ill.
Ghosh, Tushar K., Chicago, Ill.
Gibson, Robert C., East Syracuse, N. Y.
Giessen, Bill C., Cambridge, Mass.
Grieveson, Paul, Pittsburgh, Pa.
Groves, Michael T., Hamburg, N. Y.
Herman, George, Watervliet, N. Y.
Johnson, Roger N., Richland, Wash.
Kimberley, Richard H., Alabama City, Ala.
Koppenaal, Theodore J., Chicago, Ill.
Martin, Charles C., Gadsden, Ala.
Nicholas, Michael G., Bayside, N. Y.
Petit, Robert G., Wyandotte, Mich.
Proebstle, Richard A., Schenectady, N. Y.
Rueckl, Roger L., Pittsburgh, Pa.
Sharma, Kapil D., Chicago, Ill.
Stein, Dale F., Schenectady, N. Y.
Trott, Beverly D., Schenectady, N. Y.
Wanamaker, John L., Burbank, Calif.
Wells, Martin G. Headlam, Pittsburgh, Pa.
Wieser, Peter F., Niagara Falls, N. Y.

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REINSTATEMENT—JUNIOR MEMBER

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Rago, Kolli K., Los Angeles, Calif.

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Day, Robert B., Golden, Colo.

Student to Member

Scheid, Donald R., Marietta, Ohio
Schwenzfeier, Carl W., Elmore, Ohio

Student to Junior

Stanczyk, Martin H., Tuscaloosa, Ala.

SPECIAL REINSTATEMENT TO ASSOCIATE

Gibbons, Donald F., Murray Hill, N. J.

CHANGE OF STATUS

Associate to Member

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Billinghurst, Robert G., Windsor, Ont., Canada
Bitler, William R., Pittsburgh, Pa.
Cremisio, Richard S., Pittsburgh, Pa.
Gamboa, Argenis J., Caracas, Venezuela
Massobrio, Biorgio, Genova, Italy
Nanda, Madan M., Groton, Conn.
Roy, Aaron S., Murray Hill, N. J.
Stoli, Richard E., Park Forest, Ill.
Tempieton, James B., Birmingham, Ala.
Todd, Fred Allen, Anaheim, Calif.
Van Meter, Elgin, Los Angeles, Calif.
Warnes, Don E., Monaca, Pa.
Young, Kelvin A., Fort Saskatchewan, Alta., Canada

Student to Junior

Basu, Sujit K., Gainesville, Fla.

Dr. George Sachs

Dr. George Sachs, research professor and associate director of the Syracuse University Research Institute for the past eight years, died of a heart ailment at his home in Syracuse October 29. He was 64 years old.

Born in Germany, Dr. Sachs came to this country in 1936. He joined the faculty at Syracuse in 1952, and until 1959 was director of the university's metallurgical laboratories.

For his work for the US Armed Forces during World War II Dr. Sachs was awarded the Citation of Appreciation for Outstanding Contribution by the Departments of War and the Navy.

Dr. Sachs was the author of more than 300 publications and 15 books on metallurgy.

He is survived by his wife and three children.

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K. C. Li

K. C. Li, internationally known mining engineer and chairman of the Wah Chang Corp., New York, died suddenly March 7th in his New York office of a heart attack. He was 69 years old.

Known as China's unofficial ambassador to the United States, Mr. Li, a naturalized citizen, was born in Changsha, China. He was educated at the Hunan Technical Institute, China, and at the Royal School of Mines, London.

A world authority on tungsten, Mr. Li discovered the first tungsten deposits in China, and in 1915 made the first shipment of Chinese wolfram ore to this country. He served as advisor on antimony to the Allied and British Governments during World War I, and as advisor on tungsten to the US Government during World War II.

Mr. Li belonged to AIME and many other organizations.

NECROLOGY

Year Elected	Name	Date of Death
1901	Cox, Thomas (Legion of Honor)	Apr. 14, 1961
1955	Duame, Lloyd E.	Mar. 3, 1961
1905	Gordon, R. W.	Unknown
1956	Heilman, W. W.	Feb. 14, 1961
1950	Johnson, Charles W.	Unknown
1941	Johnston, James C.	Unknown
1946	Kruse, P. W.	Feb. 20, 1961
1927	Larsen, Esper S.	Mar. 8, 1961
1951	LeMaire, Edward	Dec. 11, 1960
1924	Matthews, Charles H.	February 1961
1897	Minard, Fred H.	Unknown
1914	Milward, Miles S.	Unknown
1917	Potter, George W.	Feb. 16, 1961
1940	Spindler, G. R.	Feb. 20. 1961
1957	Terrill, Ronald L.	Unknown
1921	Whitaker, Herbert	Unknown

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Technical Laboratory
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Coming Events

May 17, 1961, Joint Meeting of Powder Metallurgy Group and Physical Metallurgy Group of the New York Section, Brass Rail Restaurant, New York.

May 17-19, 1961, The Metallurgical Society, Management of Materials Research Conference, Arden House, Harriman, N. Y.

May 18-19, 1961, AIME 15th New England Regional Conference, Hotel Van Curler, Schenectady, N. Y.

May 23, 1961, Buffalo Section, NOHC, Annual Golf Party, Wanakah Country Club, Hamburg, N. Y.

June 4-9, 1961, Zinc Development Association, Sixth International Galvanizing Conference, Interlaken, Switzerland.

June 12, 1961, Pittsburgh Section, NOHC, Annual Golf Party, St. Clair Country Club.

June 14, 1961, Northern Ohio Section, NOHC, Annual Golf Party, Squaw Creek Country Club, Youngstown, Ohio.

June 17, 1961, Cleveland Section, NOHC, Annual Dinner Dance, Lake Shore Hotel, Cleveland.

June 25-30, 1961, ASTM National Meeting, Chalfonte-Haddon Hall, Atlantic City, N. J.

Aug. 7-9, 1961, Annual Conference on Applications of X-Ray Analysis, Park Lane Hotel, Denver, Colo.

Aug. 30-Sept. 1, 1961, AIME Third Technical Conference on Semiconductors, Ambassador Hotel, Los Angeles.

Aug. 31, 1961, Chicago Section, NOHC, Annual Golf Outing, Gleneagles Country Club.

Oct. 2, 1961, Chicago Section, NOHC, Fall Technical Dinner Meeting, Phil Smidt's Restaurant, Chicago.

Oct. 5-6, 1961, Southwestern Section, NOHC, Annual Fall Meeting, President Hotel, Kansas City, Mo.

Oct. 17-Nov. 7, 1961, Visit of the Iron & Steel Institute (United Kingdom) to the United States.

Oct. 20, 1961, Eastern Section, NOHC, 15th Annual Technical Conference, Warwick Hotel, Philadelphia.

Oct. 23-26, 1961, Fall Meeting of The Metallurgical Society of AIME, Pick-Fort Shelby Hotel, Detroit.

Nov. 3, 1961, Pittsburgh Section, 16th Off-the-Record Meeting, Penn-Sheraton Hotel, Pittsburgh, Pa.

Nov. 13-16, 1961, Seventh Annual Conference on Magnetism and Magnetic Materials, Hotel Westward Ho, Phoenix, Ariz.

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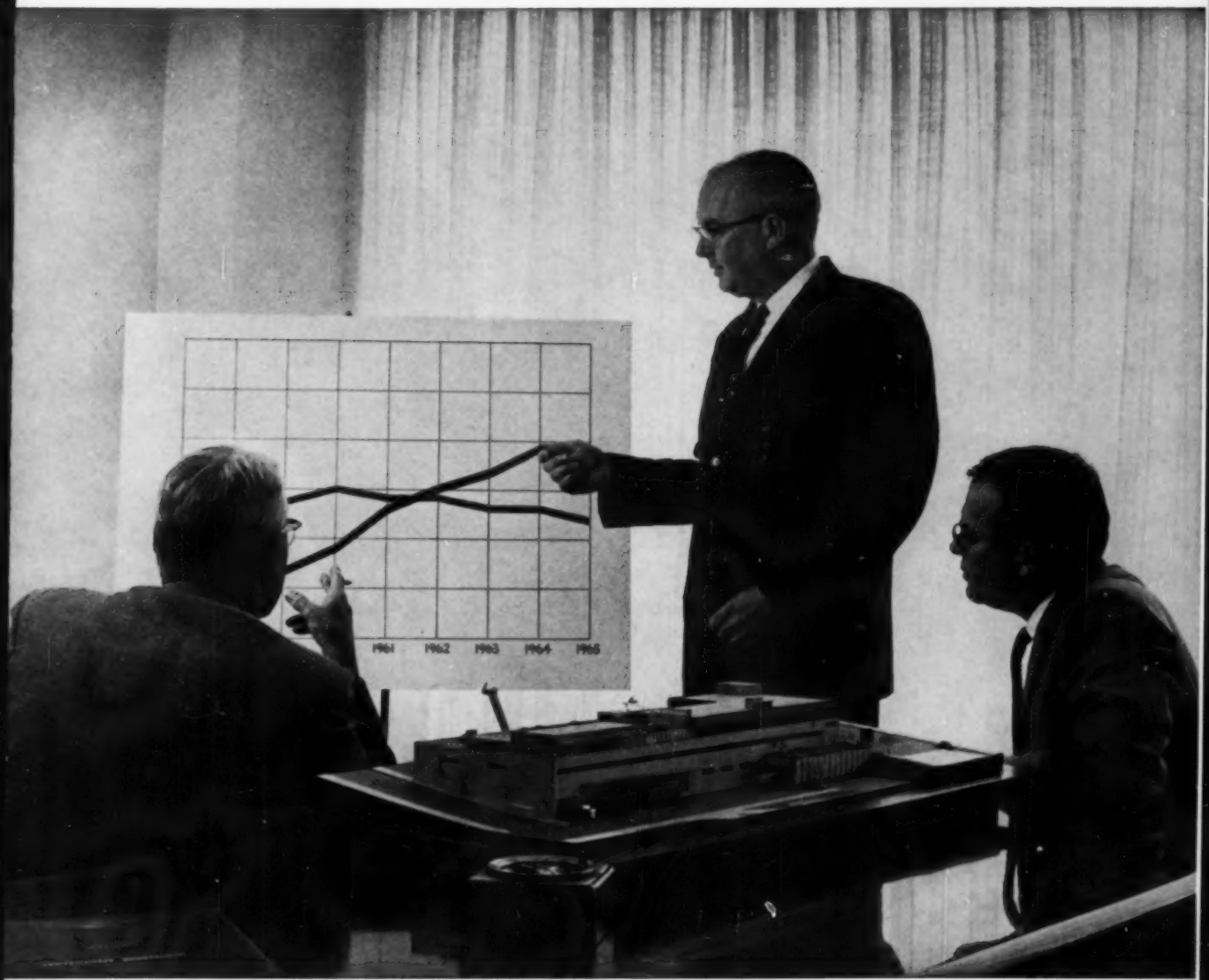

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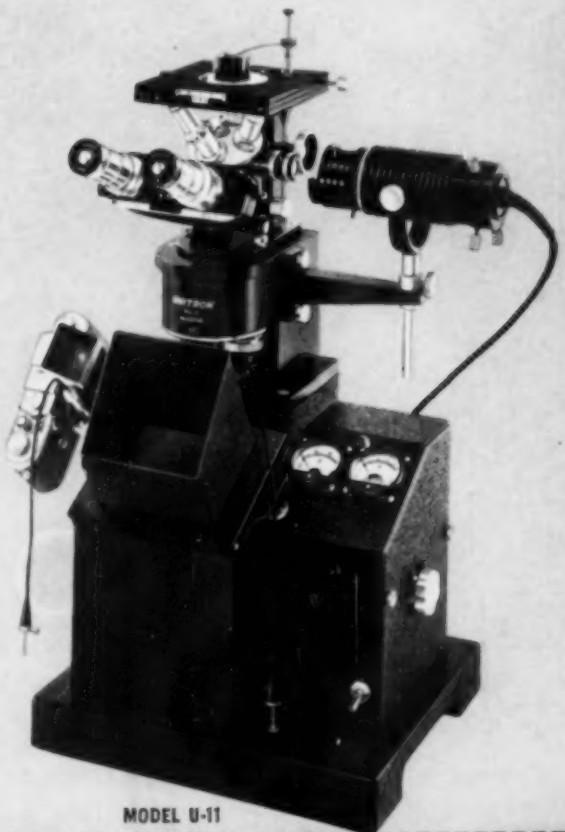

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